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White Cement Mortars Exposed

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Highlights

Characterization and Photocatalysis

Activated Charcoal in Herbal Shampoo

Discovering Thoughts, Inventing Future

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Photocatalytic Performance of White Cement Mortars Exposed in Urban Atmosphere

By Sérgio Roberto Andrade Dantas, Fúlvio Vittorino & Kai Loh

Polytechnic School of São Paulo University

Abstract- The objective of this study was evaluation of the photocatalytic performance of the addition of n-TiO₂ in white cement mortars in terms of the light reflection (evaluated by CIE L*a*b* scale) and reflectance of the mortar surfaces exposed to solar radiation in an urban environment. Are produced specimens of 1.2m × 1.2m with n-TiO₂ additions of 1%, 5%, and 10% concerning to the total dry powder mass. The performances of the specimens were then compared with two sets without n-TiO₂, painted and unpainted, and these were used as references. To conduct an in-situ experiment, the mortars were mixed and applied by a mason. After 41 months of exposure and washing of the specimens, the painted mortars and the mortars with the addition of different n-TiO₂ contents exhibited a statistically equivalent reflectance under exposure to solar radiation. All specimens exhibited color differences resulting from: (i) the accumulation of dirt on the surface; (ii) an increase in the surface roughness of the specimens; and (iii) a decrease in rainfall over the duration of exposure.

Keywords: photocatalysis, self-cleaning, solar radiation, the difference of color, urban warming.

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Photocatalytic Performance of White Cement Mortars Exposed in Urban Atmosphere

Sérgio Roberto Andrade Dantas^α, Fúlvio Vittorino^σ & Kai Loh^ρ

Abstract- The objective of this study was evaluation of the photocatalytic performance of the addition of n-TiO₂ in white cement mortars in terms of the light reflection (evaluated by CIE L*a*b* scale) and reflectance of the mortar surfaces exposed to solar radiation in an urban environment. Are produced specimens of 1.2m × 1.2m with n-TiO₂ additions of 1%, 5%, and 10% concerning to the total dry powder mass. The performances of the specimens were then compared with two sets without n-TiO₂, painted and unpainted, and these were used as references. To conduct an in-situ experiment, the mortars were mixed and applied by a mason. After 41 months of exposure and washing of the specimens, the painted mortars and the mortars with the addition of different n-TiO₂ contents exhibited a statistically equivalent reflectance under exposure to solar radiation. All specimens exhibited color differences resulting from: (i) the accumulation of dirt on the surface; (ii) an increase in the surface roughness of the specimens; and (iii) a decrease in rainfall over the duration of exposure.

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I. INTRODUCTION

Corrective maintenance is typically carried out on facades as a result of changes in their visual appearance. Is expected that a self-cleaning surface maintains its original appearance and reflectance under exposure to solar radiation to more time than the conventional coatings used in the facades of buildings.

There has been an increase in the use of facade coatings with high solar reflectance characteristics, in addition to retro-reflective materials, which reflect direct solar radiation towards the sky and not in the direction of other buildings [1-6]. An increase in the light and thermal reflectance of building facades could be an effective strategy for the reduction of urban warming, to improve indoor thermal comfort and the energy consumption of heating, ventilation, and air conditioning (HVAC) systems [7-10], which results in the mitigation of the urban heat island effect [11]. However, the constant contact between coatings and environmental degradation agents, the incidence of ultraviolet radiation [12], and the coating roughness [13] tend to

decrease the reflectance and induce changes in color over time.

The role of nano titanium dioxide (n-TiO₂) in the decontamination of water and the oxidation of several organic atmospheric pollutants as a result of photocatalytic activity well established. Moreover, although the photocatalysis not considered a reliable technology for breaking down large quantities of dirt, it can be applied to prevent accumulation [14,15]. The process is an alternative for the maintenance of surface cleanliness and clarity, in addition to constant solar reflectance, which ensures the proper operation of cooling properties [16].

Because of the large band gap and high photocatalytic activity, the n-TiO₂, it is commonly used in the photocatalysis processes. Several researchers [17-25] have investigated the addition of n-TiO₂ in its anatase mineralogical form to cement coatings, with the objective to evaluates self-cleaning surfaces upon exposure to solar radiation. Also, self-cleaning and photocatalytic materials can add market value to buildings because of the improved maintenance of the optical performance of their facades.

Krishnan et al. [26] revealed that the photocatalytic activity is significantly degraded by the presence of calcium and sulfur from the substrate, which may accumulate on the surface of the coating and penetrate the n-TiO₂ lattice, thus weakening the photocatalytic effect. The surface finish should maintain its properties over time; so, it should resist the action of environmental agents that lead to gradual erosion [14], which reduce the performance of buildings concerning the reflectance under exposure to solar radiation and the aesthetic.

So, the maintenance of color and reflectance is critical to the useful life of the buildings, and this study offers other insight into the effectiveness of photocatalytic coatings when evaluated at a long time.

II. EXPERIMENTAL PROGRAM

The tests were carried out on specimens with dimensions of 1.2m × 1.2m under exposure to an urban environment for 41 months in the city of São Paulo (Latitude: 23° 33' 15" S; Longitude: 46° 44' 1" W) in the northwest direction, to maximize exposure to sunlight, as shown in Fig.1. A slope of 33° used concerning to the ground, to obtain, (i) a higher solar radiation index; and

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(ii) to prevent the stagnation of rainwater on the specimen surfaces, thus limiting the proliferation of microorganisms.



Fig.1: Exposure of specimens

The mortars were applied by a mason, with a maximum thickness of 1 cm and with the absolute minimum roughness to obtain a high reflectance. The substrate was finished with cement paste to achieve regularisation, the homogenization of water absorption, enhanced adhesion, and to prevent an increase in the consumption of the mortars.

The reflectance under exposure to solar radiation measured for 36 months at monthly intervals. A measurement was then carried out after washing the mortars, following 41 months of exposure, to verify the restoration of the photocatalytic activity. The color measured at the beginning of the exposure time period and after 41 months of exposure, for a comparison of the initial and final color conditions. Moreover, unexposed specimens are used as references for the initial color.

a) Materials

All the mortar compositions were formulated using white Portland cement (WHITE CEM I 52.5R EN 197-1); dolomites #20, #40, and #80; an air-entraining

agent based on sodium lauryl sulfate molecules; and water-retaining agents based on cellulosic ether molecules. The n-TiO₂ used in this study was 100% anatase (ACTiVTM PC105 Ultrafine), recommended by the manufacturer for applications in the photocatalysis processes.

Twenty specimens produced, fifteen exposed to the urban atmosphere, and five used as references of the initial color. The samples were classified into five types (A, B, C, D, and E) and divided into four groups (1, 2, 3, and 4).

The mortars classified as A and B represent compositions formulated without n-TiO₂ and as a benchmark. Type-B mortars are painted, whereas type-A mortars were unpainted. The type-C, type-D, and type-E mortars were unpainted, and they represent the compositions formulated with the direct addition of different n-TiO₂ contents to the mixture. Table 1 presents the terminology and exposure conditions of the specimens.

Table 1: Composition of mortar specimens

Groups	Mortars A (unpainted) 0%TiO ₂	Mortars B (painted) 0%TiO ₂	Mortars C (unpainted) 1%TiO ₂	Mortars D (unpainted) 5%TiO ₂	Mortars E (unpainted) 10%TiO ₂	Exposure
1	A1	B1	C1	D1	E1	Exposed
2	A2	B2	C2	D2	E2	
3	A3	B3	C3	D3	E3	
4	A4	B4	C4	D4	E4	Unexposed

i. *Raw Material Characteristics*

Table 2 shows the characteristics of the raw materials, in accordance with a study by Dantas et al. [27]. For determination of the specific surface area (SSA), the Brunauer–Emmett–Teller (BET) method was employed, and the real density analysis was determined using the He pycnometer method. The particle size distribution of finer particles was determined using laser

granulometry, and the dolomite particle size distribution was determined using a dynamic image analyzer.

The mineralogical compositions of the white Portland cement (WPC) and n-TiO₂ were determined by X-ray diffraction using the Rietveld analysis method, and the chemical composition of the cement determined by the Brazilian Association of Technical Standards (ABNT) and ASTM standards.

Table 2: Raw material characteristics

Materials	Diameter (µm)			Specific surface area (m ² /g)	Average Density (cm ³)
	d ₁₀	d ₅₀	d ₉₀		
White Portland cement	2.6	17.7	19.5	0.86	3.05
Dolomite #20	975.1	1242.1	1620.5	0.16	2.90
Dolomite #40	24.3	230.0	739.6	0.56	2.94
Dolomite #80	4.5	38.3	133.9	0.80	2.81
n-TiO ₂	0.66	1.50	4.59	79.8	3.62

ii. *Mortar specimens composition*

Each composition was prepared with a different n-TiO₂ (1%, 5%, and 10%) and water contents to ensure the same workability for all, as defined by the experience

of the mason during the mixing. This procedure was adopted to conduct an in-situ experiment. Table 3 shows the consumption of each raw material.

Table 3: Consumption of each raw material (kg/m³)

Materials	Mortars A (unpainted) 0%TiO ₂	Mortars B (painted) 0%TiO ₂	Mortars C (unpainted) 1%TiO ₂	Mortars D (unpainted) 5%TiO ₂	Mortars E (unpainted) 10%TiO ₂
White Portland Cement	117.6	117.6	116.1	106.9	99.2
Dolomite # 20	68.7	68.7	67.9	62.5	58.0
Dolomite # 40	386.6	386.6	381.8	351.5	326.1
Dolomite # 80	106.4	106.4	105.0	96.7	89.7
Water retained	1.5	1.5	1.5	1.4	1.4
Air-entrainment	0.2	0.2	0.2	0.1	0.1
Water	319.0	319.0	322.1	355.9	379.2
n-TiO ₂	---	---	5.4	25.0	46.3

III. METHODS

a) *Measuring reflectance indices*

The reflectance values were determined in accordance with the methods given in the ASTM and ASHRAE standards [28,29]. All measurements were carried out from 11h00 to 13h00 because of the highest incidence of global solar radiation on the specimens surfaces. The measurements were carried using two pyranometers, with measuring ranges of 305–2800 nm and maximum measuring ranges of up to 2000 W/m², with an output signal of 0–50 mV and sensitivity of 10–35µV/W/m². A data acquisition system was employed using a datalogger with a 6.5-digit display and

conversion rate (maximum) of 10 measurements per second, with an RS232 output. An acquisition rate of 1 measurement per second and acquisition time of 20 s are employed, by a previous study by Dantas, Vittorino, and Loh [12]. Fig.3 presents the measurement procedure.

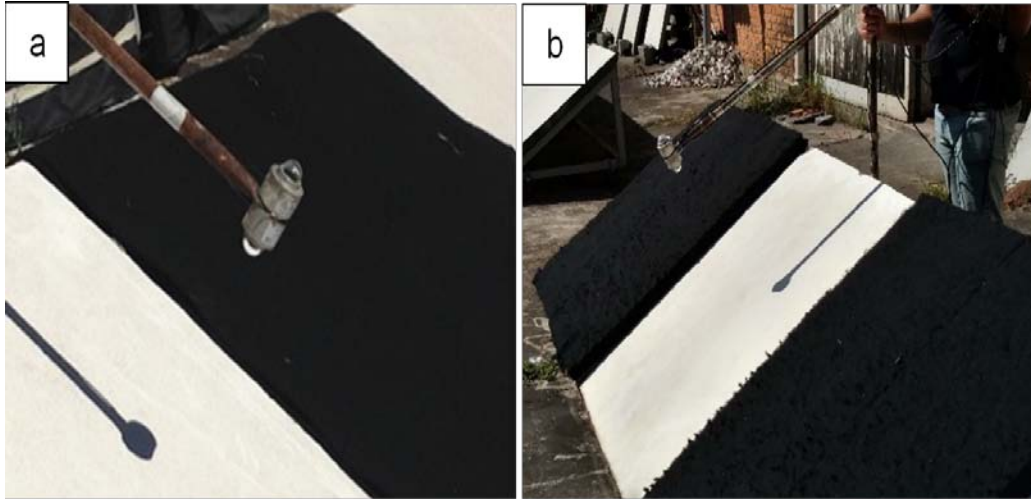


Fig. 3: (a) Pyranometers and (b) data collection

b) Measurement of color

The evaluation of the color differences and yellowing index (YE) was carried out by the ASTM standard [30], using a Spectro-Guide Sphere $d/8^\circ$ spin spectrophotometer with geometric dimensions of 45 circ./0, $d/8$. A measurement area within the range of 400–700nm, the spectral resolution of 20 nm, photometric area of 0–100% (0.01), and standard observer D65 with an aperture angle of 10° were employed. The measuring procedure was carried out as previously described by Dantas, Vittorino, and Loh[12].

c) Optical microscopy analyses

Surface samples were obtained from the specimens and stored in plastic bags. No preparation process was carried out on the samples before, to ensure the maintenance of the as-exposed state. The surface textures of the mortars and the surface $n\text{-TiO}_2$ dispersions were observed using an Eclipse electronic microscope with a 40-fold increase, a fibre optic illuminator, and a digital camera with a resolution of 3.2 megapixels.

specimens exhibited a lower roughness than those of the other specimens in the early stages of the exposure time period. Also, a lower rugosity can prevent impregnation and to ease the removal of dirt by rainfall, resulting in an increased reflectance under exposure to solar and luminous radiation. However, this is not observed after 24 months of exposure, when the acrylic film exhibited degradation, allowing for increased accumulation of dirt on the surfaces of type-B specimens. Fig.4 presents the surfaces of the mortars.

IV. RESULTS AND DISCUSSION

Observation of the samples over the exposure period revealed a direct relationship between the solar and luminous reflectance, rainfall incidence, and roughness of the samples. An increase in the roughness of the mortars over the exposure time period was observed, which allowed for an increase in the accumulation of dirt on the specimens. In combination with the low rainfall during the first year of exposure, this resulted in a higher impregnation of the samples by dirt.

Visual inspection using an optical microscope revealed an increase in the roughness of the specimens surfaces. The images revealed that the type-B

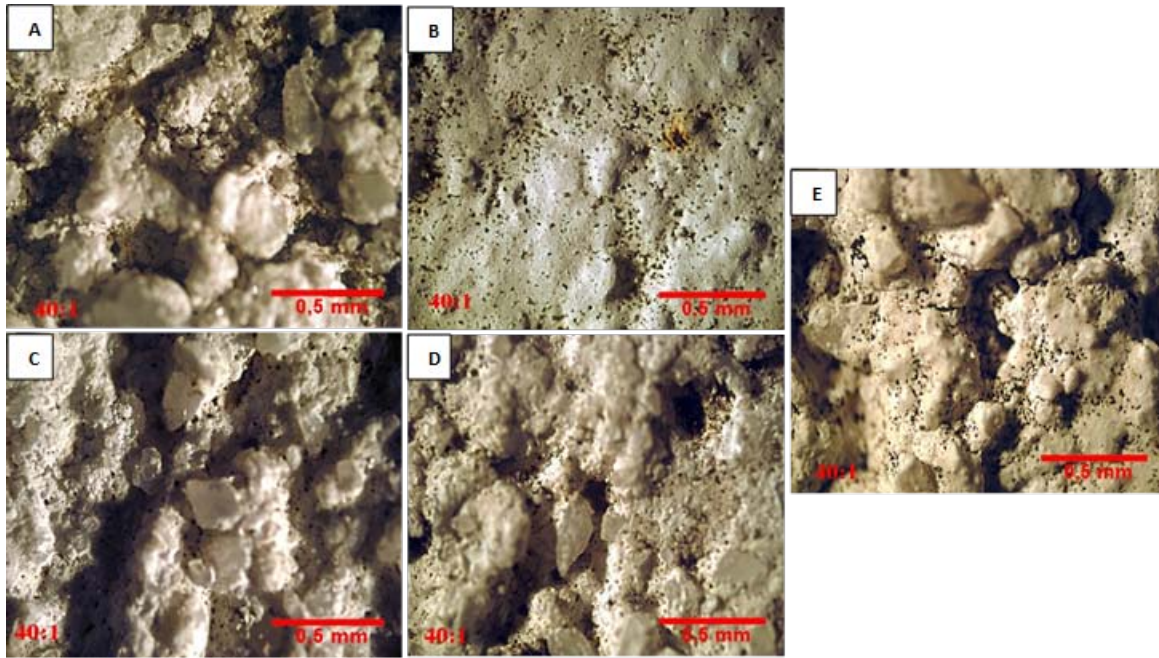


Fig. 4: The surface roughness of specimens

a) Solar reflectance results

The mortars exhibited similar behaviors with respect to solar radiation reflectance. Therefore, the results for each mortar evaluated can be presented concerning to the mean reflectance, as suggested by Dantas, Vittorino, and Loh [12] and Dantas; Vittorino [31].

Fig.5 presents the mean (average of three values of each sample) reflectance of the specimens over the 41 months of exposure to the urban environment. Figs.6 and 7 present the total global solar radiation incident on the horizontal surfaces and the total monthly rainfall in São Paulo city, respectively.

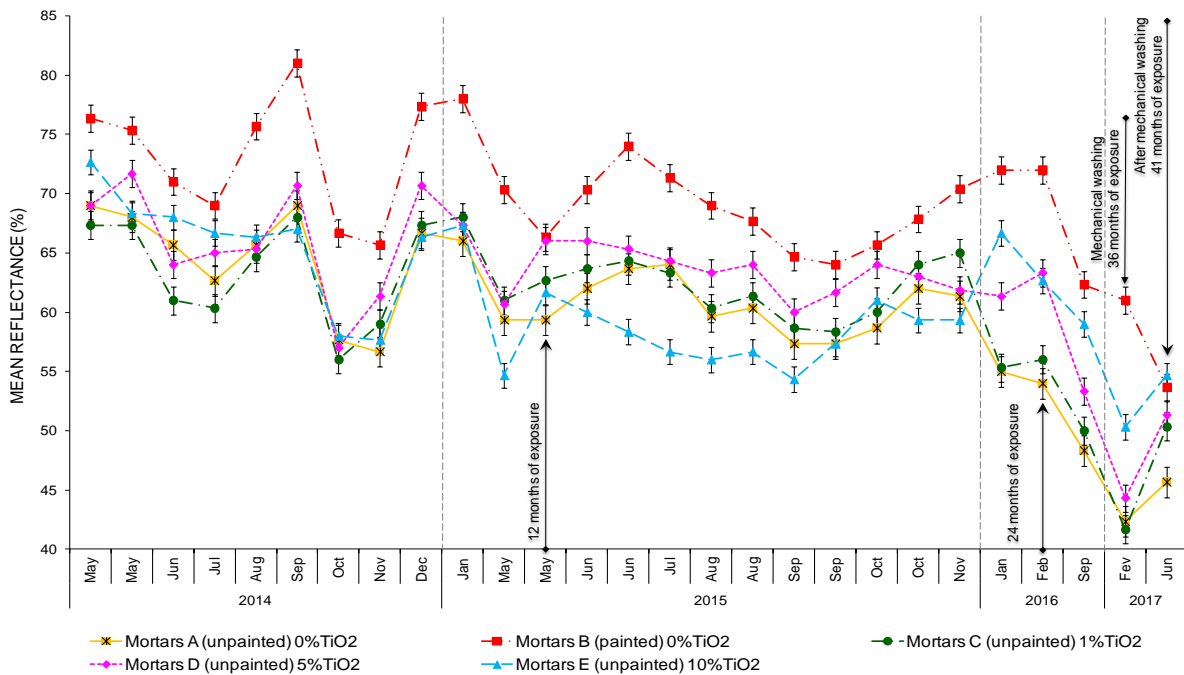
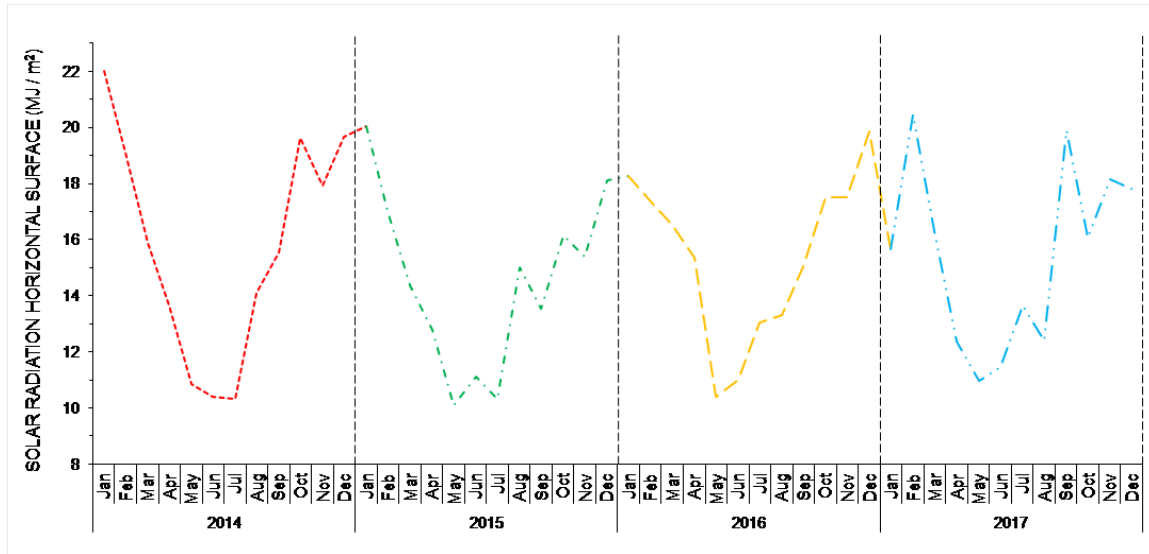
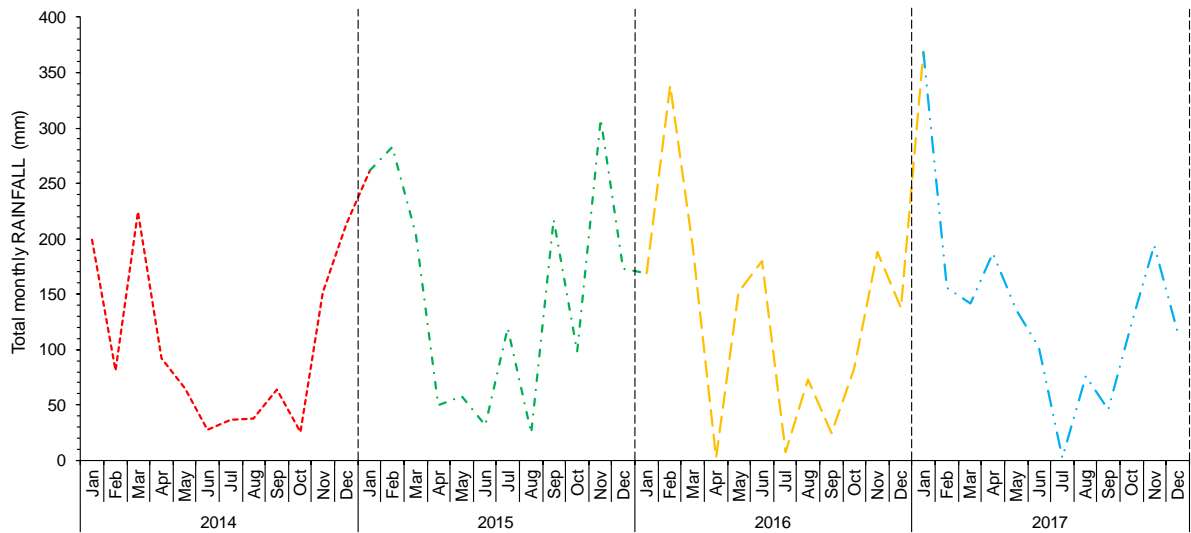


Fig. 5: Mean reflectance of mortars after 41 months of exposure to an urban environment



Source: Institute of Astronomy, Geophysics and Atmospheric Sciences

Fig. 6: Solar radiation on horizontal surfaces in São Paulo city



Source: Institute of Astronomy, Geophysics and Atmospheric Sciences / National Institute of Meteorology

Fig. 7: São Paulo city monthly rainfall

During the first 12 months of exposure, type-B specimens exhibited a noticeable high reflectance, given that the acrylic paint did not exhibit degradation. Moreover, no marked differences are observed among the reflectance results of the other mortars groups, which could be considered to have equal values. For type-A mortar, a decrease in the reflectance was expected as a result of the natural aging process, which results from the accumulation of dirt. For types C, D, and E, their initial reflectance values were expected to remain stable for a longer time period. Also, the small

differences observed between their reflectance values were due to the different n-TiO₂ contents. However, this behavior was not noticed after one year of exposure because of the lack of rainfall and high impregnation of the specimens surfaces by dirt.

After 12 months, type-E mortars exhibited a decrease in the reflectance under exposure to solar radiation when compared with the other mortars. This difference remained significant until the 16th month of exposure. However, with longer exposure time, an important difference in the behavior of the mortars was

observed. The increase in reflectance from September (2015) to October (2015) was directly related to the high rainfall that occurred during this period (Fig.7) and the increase in solar radiation (Fig.6) associated to the beginning of the spring season. This two factors contributed to the cleaning of the specimens surface and the increase in the photocatalytic activity during this period.

From October (2015) to January (2016), the seasonal changes caused reflectance changes on the mortars surface. At the beginning of the summer season, there was an increase in the incidence of solar radiation and a decrease in rainfall. Because of this, different effects occurred on the mortars surfaces, that can be attributed to the variation of precipitation, the surfaces roughness, and their levels of dirt impregnation.

In the second year (2016), considerable differences in the values of the reflectance under

exposure to solar radiation were observed in all specimens. From February (2016) to February (2017), the mortars exhibited a continuous and significant decrease in reflectance. This behavior can be attributed to the low rainfall over these two years, which resulted in the impregnation of the specimens surfaces by dirt.

In the third year (2017), a significant decrease was observed in the values of reflectance under exposure to solar radiation in all the specimens. Over the three years of exposure, apart from type-B samples, all the mortars exhibited a higher impregnation by dirt, which limited the photocatalytic activity. After that, the samples were cleaned using a washing machine, to reproduce the process commonly employed for cleaning building facades. Fig.8 presents some examples before and after the washing.



Fig.8: (a) Before washing; (b) during washing; (c) difference between specimens; and (d) after washing

After washing (4st month) and a short period of exposure, a reflectance measurement was carried out. The main objective was to verify the restoration of the photocatalytic activity and the initial color. The final measurement, after the washing, revealed that the reflectance of type-B specimens under exposure to solar radiation are not restored. This loss of reflectance under exposure to solar radiation can be attributed to the degradation of the paint film, which resulted in the exposure of the mortar to a higher impregnation by dirt. Nevertheless, after washing, all the other mortars exhibited a restoration concerning their reflectance under exposure to solar radiation.

The addition of different n-TiO₂ contents to the mortars did not result in statistical differences between

the characteristics of reflectance under exposure to solar radiation after washing. These results reveal that the effectiveness of the photocatalytic process of the mortars is not dependent on the added n-TiO₂ content.

b) Color change results

For evaluation of the white color, the CIE L*a*b* components (ΔL^* / Δa^* / Δb^*) to be individually considered for a better perception of the changes in the shades of the mortars. The components were calculated using simple arithmetic differences, and Fig.9 presents the initial values and the differences between the colors at the beginning of exposure and after 41 months of exposure, following the washing of the specimens.

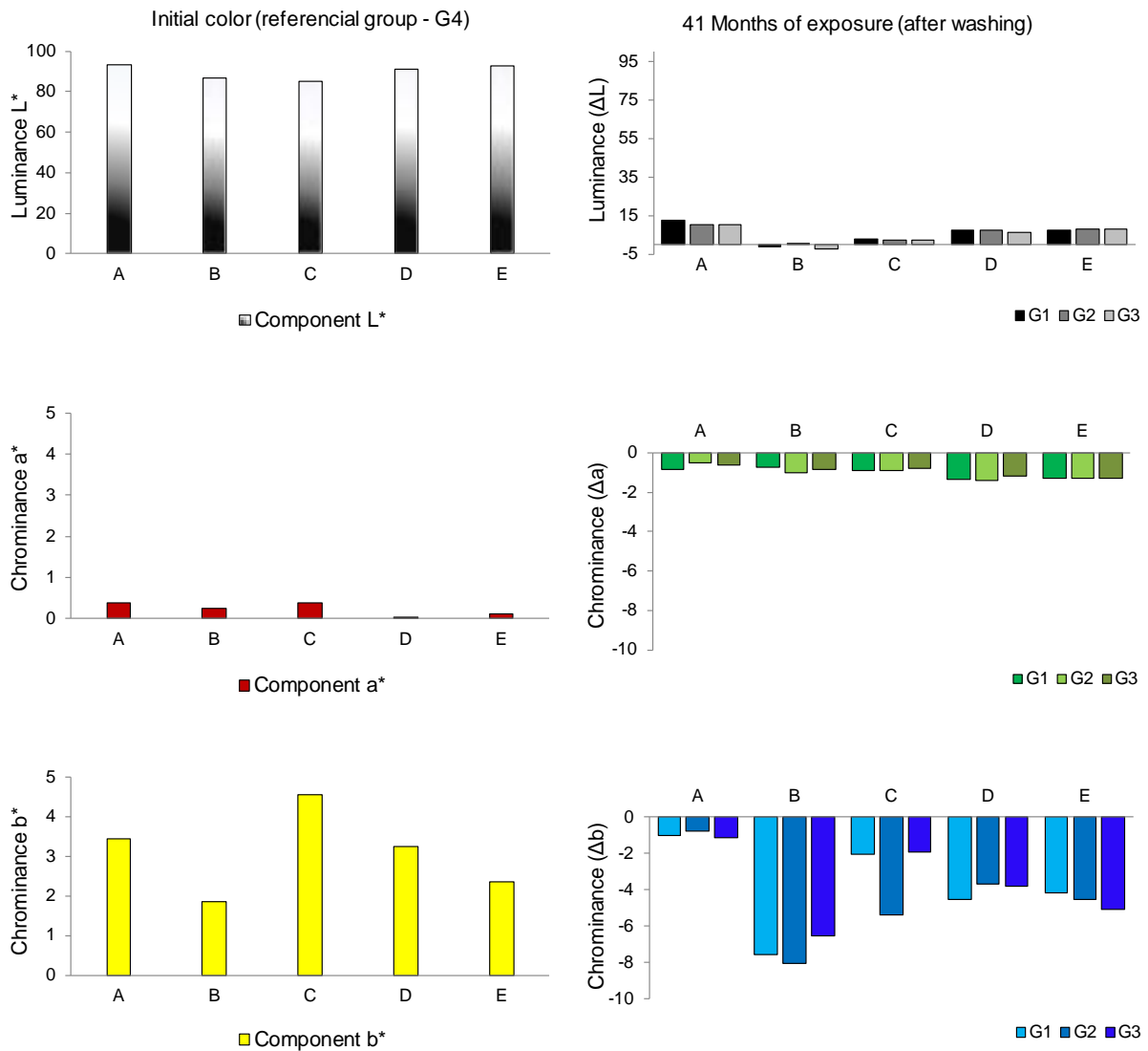


Fig. 9: Changes in color of mortars by CIE L*a*b*

After 41 months of exposure and washing, is not observed significant differences between the luminance (ΔL) values of type-B and type-C mortars. Type-A, type-D and type-E mortars exhibited more noticeable differences in color concerning to luminance (ΔL). This behavior is expected for type-A mortars, which were not subject to pre-treatment (e.g., painting or water repellent), thus allowing for a higher deposition of dirt on the surface.

For the type-D and type-E mortars, the photocatalytic activity was expected to be more effective because of the higher levels of n-TiO₂ contents used in the mixtures. However, no differences observed between the mortars concerning luminance (ΔL), which indicates that an increase in the added n-TiO₂ content does not influence the photocatalytic activity. Besides, the incidence of solar radiation and the amount of rainfall on the specimens has an impact on the photocatalytic activity. As previously highlighted, the

ineffectiveness of the photocatalytic activity can connect to: (i) the increased accumulation of dirt on the specimens surfaces; (ii) increases in the surface roughness of the mortars, and (iii) the impact of n-TiO₂ non-dispersion that resulted in the formation of agglomerates, as shown in Fig.10.

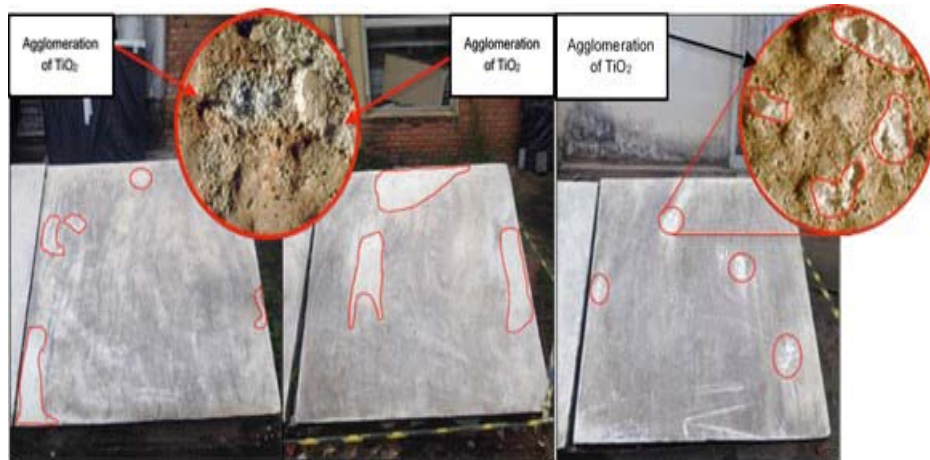


Fig.10: n-TiO₂ agglomerates of E-type mortars

From the evaluation of the luminance (ΔL), observes a significant color difference between type-B and type-A (unpainted and without n-TiO₂), type-D, and type-E mortars (higher n-TiO₂ content). When compared with type-C mortar (low n-TiO₂ content), no observes marked color differences. Moreover, concerning to the chrominance components (Δa and Δb), color differences were observed in all mortars.

From the evaluation of the Δa component (green and red), no noticeable color differences were observed between the all mortars. These results indicated that with respect to this chromatic component, there were no significant differences between type-B specimens and the other mortars. The Δb component (blue and yellow) exhibited the most significant color differences. Is observed marked differences in type-B specimens, which was in good agreement with the measurements of the reflectance under exposure to solar radiation, as a result of the degradation of the acrylic resin in the paint composition.

In general, type-A mortars exhibited the least significant difference concerning to chrominance. This behavior is associated with the high deterioration of specimen A1, which led to the displacement of the mortar, thus altering the general data.

Among the mortars with the added n-TiO₂ contents, type-C mortar exhibited the least significant color difference; where as type-D and type-E mortars exhibited the same degree of color difference, which was more significant than that of type-C mortar.

Thus, concerning to the difference of color (measured according to CIE L*a*b* scale), differences in the chromatic components of the mortars were observed upon evaluation after 41 months of exposure, following the washing, as shown in Fig.9.

- a) Type-A mortars exhibited more significant color perception differences concerning to luminance (ΔL), and lower chrominance (Δa and Δb).
- b) Type-B specimens exhibited less significant color perception differences concerning to luminance (ΔL)

and more significant color perception differences concerning to the chrominance component Δb . Moreover, no observes significant color perception differences concerning to the chrominance component Δa .

- c) Type-C mortars exhibited less significant color perception differences than type-B specimens concerning to luminance (ΔL). Moreover, observed a less significant color difference in the chrominance component Δb with those of type-D and type-E mortars. No observes significant color differences in the chrominance component Δa .
- d) Type-D and type-E mortars exhibited more significant color perception differences concerning to the luminance (ΔL) and the chrominance component Δb . However, no observes significant color difference in the chrominance component Δa .

The yellowing index (YE) determined by the spectrophotometer (see Fig.11) was higher for type-B specimens than it was for the other. The yellowing of the specimens was also observable using the naked eye when compared with other white-color surfaces. This change was due to the degradation of the acrylic film of the paint over the long-term exposure.

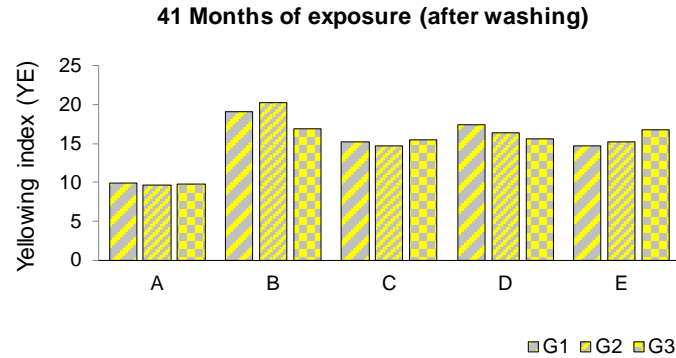


Fig. 11: Yellowing index (YE) by CIE L*a*b*

It is reasonable to use ΔE as a measure of the difference between the visual appearances of two given colors. Although the results indicated by ΔE can be used to determine color differentiation, this should only be considered as a general guide, as it is possible to obtain a ΔE value of less than 1.0 for two colors that appear different.

The definition of ΔE differs slightly depending on the formula used, which indicates that it may not always be a reliable measure. Hence, observations made by the human eye may be required to verify the final answer, and more significantly, to provide a delimitation of the acceptable minimum and maximum limits for a specific application.

Considering that human eyes are more sensitive to luminance than chrominance, it may appear as though a surface has lost luminance (ΔL), when it has instead lost chrominance (Δa and Δb), which ranges from green to red or blue to yellow colors, resulting from the presence of fungi or soot.

After washing all the specimens exhibited white color surfaces, in the observation by the naked eye. However, in comparison with the colors in the beginning, different shades were observed for the specimens. Considering that the perception of color change is intrinsically subjective and dependent on the personal judgment of each observer, the perception of the white color is exclusively dependent on the type of lighting in which the surfaces are evaluated, in addition to the intensity of the light. The specimens exposed into an urban environment, which implies that there were variations in the visual perception of the observer concerning the time of day, degree of occurrence, and amount of rainfall during the surface evaluation. However, the spectrophotometer indicates the same color, independent of the weather conditions. Fig. 12 presents the differences in the color perceptions of the mortars at the beginning ($t = 1^{\text{st}}$ month) and end ($t = 41^{\text{st}}$ month) of the exposure time period.



Fig.12: Color perception of specimens: (a,b, and c) 1st month and (d,e, and f) 41st month

This differentiation is associated with two factors, as follows.

- The chromatic components (Δa and Δb), which contributed significantly to the results obtained using the spectrophotometer, as the loss of the initial white color.
- The light reflected by the surface of the specimens, which results in a phenomenon referred to as metamerism. Because of the limitations of the spectrophotometer, this phenomenon was not detected using the equipment.

V. CONCLUSIONS

The use of specimens with larger sizes (1.2m \times 1.2m) facilitated the analysis of the photocatalytic effect with n-TiO₂, as well as the color differences. It was possible to observe the influences that are generally unobservable when small specimens are employed; such as the influence of the mason during application, the in-situ mixing process, the pollution in the accumulation of dirt, and the heterogeneity of the specimens, which typically occurs during the application of mortars.

The results obtained in this study therefore revealed the following.

- After three years, the mortars with higher n-TiO₂ contents exhibited a reflectance statistically

equivalent to that of the other mortars, under exposure to solar radiation. This indicates that the effectiveness of the photocatalytic mortars is not dependent on the added n-TiO₂ content.

- Concerning to the color differences (measured according to the CIE L*a*b* scale), there was a significant difference between the luminance (ΔL) of type-B specimens (painted and without n-TiO₂) and those of type-A (unpainted and without n-TiO₂), type-D, and type-E (higher n-TiO₂ content) mortars. Concerning to type-C mortars (low n-TiO₂ content), no observes significant color differences. However, observes increase color differences in all specimens about the chrominance components (Δa and Δb).
- The rugosity of the surfaces and the rainfall indices influenced the self-cleaning effect throughout the study.
- A vital thing observed is that the evaluations carried out within a short period of exposure to an urban environment did not reveal the effectiveness of the added n-TiO₂. So, higher exposure time to be necessary to determine the differences concerning the maintenance of the initial conditions of the mortars with added n-TiO₂ contents, when compared with the painted mortars.

ACKNOWLEDGEMENTS

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Studies on the Addition of Activated Charcoal in Herbal Shampoo

By K. Hema Divya

Abstract- The aim was to formulate a shampoo containing activated carbon which clears sebum, dirt, dandruff in hair. In this shampoo activated charcoal was added for effective removal of dust and toxic substances. Natural ingredients was used to prepare this shampoo, an emphasis on safety and efficacy, which will avoid the risk posed by chemical components. The herbs like Sikakai, Reetha, Aavaram Poo, Thulasi, Nannari, VettiVerr, Lemon Skin, Rose Flower, Kuppaimeni, Amala, Hibiscus, Mint, and Meethi have was selected to formulate the herbal shampoo. In recent times, the use of activated charcoal has become a most sought method for various purification techniques based on adsorption. It was used as one of the ingredients in cosmetics, toothpastes, and brushes. Activated charcoal also finds application in health care technologies. The shampoo was prepared and tested for various parameters such as physical appearance, pH, and percentage of solid contents, dirt dispersion, cleaning action, surface tension, and detergency ability.

Keywords: *activated carbon, dandruff, dirt, herbal shampoo.*

GJRE-C Classification: *For Code: 090499*



Strictly as per the compliance and regulations of:



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Abstract- The aim was to formulate a shampoo containing activated carbon which clears sebum, dirt, dandruff in hair. In this shampoo activated charcoal was added for effective removal of dust and toxic substances. Natural ingredients was used to prepare this shampoo, an emphasis on safety and efficacy, which will avoid the risk posed by chemical components. The herbs like Sikakai, Reetha, Aavaram Poo, Thulasi, Nannari, VettiVerr, Lemon Skin, Rose Flower, Kuppaimeni, Amala, Hibiscus, Mint, and Meethi have was selected to formulate the herbal shampoo. In recent times, the use of activated charcoal has become a most sought method for various purification techniques based on adsorption. It was used as one of the ingredients in cosmetics, toothpastes, and brushes. Activated charcoal also finds application in health care technologies. The shampoo was prepared and tested for various parameters such as physical appearance, pH, and percentage of solid contents, dirt dispersion, cleaning action, surface tension, and detergency ability.

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I. INTRODUCTION

Hair was one of the external barometers of internal body conditions. It was an important part of the human body. Various synthetic compounds, chemicals, dyes, and their derivatives have been proved to cause harmful aftermath. Nowadays, people are having an awareness of their effects on hairs skin and eyes. Due to these reasons, the community was attracted to herbal products due to their inexpensive nature and negligible side-effects. Herbal cosmetics were denoted as products formulated using various permissible cosmetic ingredients to form the base in which one (or) more herbal ingredients were used to provide accurate benefits. Nowadays, the usefulness of herbs in the cosmeceutical production had extensively increased, and there was a demand for herbal cosmetics. As far as the herbal shampoos were concerned on the nature of the ingredients, they may be simple shampoo, antiseptic (or) antidandruff shampoo and nutritional shampoo containing vitamin, amino acids, proteins hydrolysate. The selection of active components for hair care were based on the ability of the ingredients to prevent skin damage as well as to improve the quality of skin by cleansing, nourishing and protecting the skin. In this study, the formulation and evaluation of herbal shampoo powder are reported.

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The scope of the present research work was to develop an herbal shampoo powder with activated charcoal which clears sebum, dirt, dandruff, promotes hair growth, and strengthens hair. Moreover, it also acts as a conditioning agent. This herbal shampoo powder performs all these actions without affecting or damaging hair.

II. METHODS AND MATERIALS

a) Sample collection

Different parts of the plant are selected to study its hair care property. The plants used are Sikakai, Reetha, Aavaram Poo, Thulasi, Nannari, VettiVerr, Lemon Skin, Rose Flower, Kuppaimeni, Amala, Hibiscus, Mint, and Meethi. All the required powders were collected from the local herbal drug store market. The raw materials collected were given with their uses below.

Shikakai (*Accaciaconcina*)

- Cleanser
- Makes hair silky and smooth
- Treats scalp related diseases
- Anti-dandruff agent

Amla (*Phyllanthus emblica*)

- Prevents graying of hair
- Helps to increase hair growth
- Prevents lice

Mint (*Menthapiperita*)

- Promotes hair growth
- Supports to neutralize the aftermath of excessive oil Production
- Hydrates hair follicles

Hibiscus (*Hibiscus rosa-sinensis*)

- Revives dead hair follicles
- Promotes strong and healthy hair
- Makes hair root strong

Reetha (*Sapindusdetergens*)

- Provides shiny and silky hair
- Gives a cool feel on the scalp

Avaram Poo (*Sennaauriculata*)

- Keeps the body cool
- Cleanser

Vetti Ver (*Chrysopogonzizanioides*)

- For fragrance

- Lemon Skin (*Citric acid*)
- Anti-dandruff
- Provides shiny hair

Fenugreek Leaves (*Trigonellafoenum-graecum*)

- Anti-dandruff
- Keeps the body cool

Kuppai Meni (*Acalyphaindica*)

- treats dandruff

Thulasi (*Ocimumtenuiflorum*)

- Makes hair feel fresh
- Prevents hair loss
- Prevents dry scalp
- Boosts immunity of hair

Rose Powder (*Rosaceae*)

- A natural moisturizer that promotes hair growth
- Improves circulation of blood
- Hair becomes soft and smooth

Nannari (*Hemidesmusindicus*)

- Perfume
- Hair growth
- Activated Charcoal
- Removes toxicity
- Prevents dandruff formation
- Prevents graying of hair

III. FORMULATION OF HERBAL SHAMPOO POWDER

These powders were accurately weighed, passed through sieve No. 100 and then mixed in their ascending order of quantities with continuous trituration and stored in airtight containers until it was used for further studies. Three batches of the herbal shampoo powder formulations (1-3) are prepared, labeled and stored in a well-closed container and used for further studies. The preparation formulas were given in Table 1.

Table 1: Constituents and their composition

S. No	Constituents	Sample 1 (gm)	Sample 2 (gm)	Sample 3 (gm)
1	Shikakai (<i>Accaciaconcina</i>)	16.67	16.67	16.67
2	Amla (<i>Embliaofficinalis</i>)	3.33	3.33	3.33
3	Mint (<i>Menthapiperita</i>)	3.33	3.33	3.33
4	Hibiscus (<i>Hibiscus rosa-sinensis</i>)	2.5	2.5	2.5
5	Reetha (<i>Sapindusdetergens</i>)	6	6	6
6	Avaram Poo (<i>Sennaauriculata</i>)	3.33	3.33	3.33
7	VettiVer (<i>Chrysopogonzizanioides</i>)	3.33	3.33	3.33
8	Lemon Skin (<i>Citric acid</i>)	1.34	1.34	1.34
9	Fenugreek Leaves (<i>Trigonellafoenum-graecum</i>)	3.33	3.33	3.33
10	KuppaiMeni (<i>Acalyphaindica</i>)	3.33	3.33	3.33
11	Thulasi (<i>Ocimumtenuiflorum</i>)	3.33	3.33	3.33
12	Rose Petals (<i>Rosaceae</i>)	1.34	1.34	1.34
13	Nannari (<i>Hemidesmusindicus</i>)	1.34	1.34	1.34
14	Activated Charcoal	3.5	2.5	1.5

IV. EVALUATION OF HERBAL SHAMPOO

a) Organoleptic evaluation

Color

The color of the herbal shampoo was observed and noted.

Odor

The odor of the shampoo was observed.

Texture

The texture of the shampoo was observed.

b) Physiochemical Evaluation

pH

5% shampoo solution was taken and the pH was determined using a pH meter at room temperature.

Active matter

2.2 gm of the sample was weighed accurately, and it is made up to 250 ml by addition of distilled water in a standard flask. It was shaken well and allowed to

$$\text{Anionic active matter, percent by mass} = (342 \times V \times M \times 5) \div m$$

where,

342-Molecular mass of sodium alkyl benzene sulphonate taken for calculations

V-Volume in ml of benzethonium chloride solution added

M-Molarity of benzethonium chloride solution

m-Mass in g of the sample taken

Non-volatile alcohol-soluble matter

2 gm of the sample was taken, and 50 ml of 95 % ethanol was added to it. The sample was soaked in

$$\text{Percentage of non-volatile alcohol soluble matter} = (X - Y \times 100) \div z$$

where,

x - Weight of the sample

y - the initial weight of the empty crucible

z - the final weight of the crucible with residue

Foam Height

2 gm of the sample was weighed accurately, and mixed in 100 ml of distilled water. The mixture was taken in a 500 ml graduated cylinder, and it was shaken for 50 times. The test was carried at room temperature. The foam was formed due to the foaming ability, and its height was noted.

Foam Stability

In Water Base

2 gm of the sample was weighed accurately, and mixed in 100 ml of distilled water. The mixture was taken in a 500 ml graduated cylinder, and it was shaken for 50 times. The test was carried at room temperature. Foam was formed due to the foaming ability. The formed foam retention time was noted.

dissolve for some time. After a few minutes 10 ml of the sample was pipetted out. Standardized benzethonium chloride solution was filled in the burette. 0.05 gram of methylene blue, 50 grams of sodium sulfate and 68 ml of concentrated sulphuric acid was dissolved in water; methylene blue solution was prepared. This methylene blue solution was used as an indicator. This methylene blue solution was added to the pipetted out sample. 0.5 ml of benzethonium chloride solution was added slowly from the burette. The cylinder was stoppered and shaken well after each addition. The phases were allowed to separate. Initially, the chloroform layer was colored blue or greenish blue. Towards the end, the color started to migrate to the aqueous layer. The color intensity in both the phases were the same when viewed under standard conditions of light was noted.

The anionic active matter as sodium alkyl benzene sulphonate was calculated as

the solution for 5 minutes. The mixture was then heated in a water bath for 15 minutes. After some time when all the alcohol-soluble matter was dissolved, the solution was filtered to remove the remaining insoluble matter. The empty weight of the crucible and along with the residue was observed. This mixture was allowed to dry for 1 to 2 hours in an autoclave at 95 °C. The final weight was recorded after drying. It was calculated as

In Ethanol Base

0.3 gm of the sample was weighed. The sample was added to an ethanol solution (99.9% ethanol 5 ml, and water 25 ml). The mixture was taken in a 100 ml graduated cylinder, and it was shaken for 20 times. Foam was produced. The foam retention time was noted.

Dirt Dispersion

A pinch of shampoo was taken in a large test tube. 10 ml of distilled water, and one drop of India Ink (blue ink) was added to the mixture. The test tube was stoppered and shaken for ten times. The amount of ink in the foam was estimated as (None, Light, Moderate, or Heavy), and recorded.

Microbiological Assay

The yeast and mold was cultured for five days. The samples with various carbon proportions were added to these cultures, and their reduction count was noted after two days. The base count is 12,00,000 CFU/gm.

Critical Micelle Concentration

The sample was weighed, and taken in five different proportions. 50 ml of distilled water was mixed in each. This mixture was taken in a measuring cylinder, and was shaken 20 times. Foam was produced in various heights for various concentrations. These heights were measured and the graph was plotted for concentration vs. foam height. Initially, for increment in concentration, the foam height was also increasing. After a particular volume of solution, the foam height was found to be decreasing. The maximum foam height achievable was found from the graph. The corresponding concentration was the Critical Micelle Concentration (CMC) for the sample.

V. RESULTS

a) Organoleptic evaluation

The outcome of visual inspection for all the shampoo powders were observed and evaluated for color, odor, taste, and in terms of their appearance, flow property, and texture. They showed a distinct change in color. The results were reported in Table 2.

Table 2: The evaluation parameters of organoleptic studies

Evaluation Parameters	Sample 1	Sample 2	Sample 3
Colour	Greenish Grey	Greenish Grey	Greenish Grey
Odor	Slight	Slight	Slight
Texture	Fine	Fine	Fine

b) Physiochemical evaluation pH

The composition of activated carbon did not alter the pH of the shampoo. pH was noted as 5.5 which

lies within the required standards of IS 7884:2004. The result was reported in Table 3.

Table 3: The evaluation of pH

SAMPLE NO.	RESULT	UNIT
Herbal shampoo 1	5.50	-
Herbal shampoo 2	5.52	-
Herbal shampoo 3	5.56	-

c) Active matter

The composition of activated carbon did not alter the parameter of the shampoo. 9.6 g/100 g was the noted value which lies within the required standards of IS 4955:2001. The result was reported in Table 4.

Table 4: The evaluation of the active matter

Sample No.	Result	Unit
Herbal shampoo 1	9.65	gm/100 gm
Herbal shampoo 2	9.67	gm/100 gm
Herbal shampoo 3	10.50	gm/100 gm

d) Non-volatile alcohol-soluble matter

The composition of activated carbon did not alter the non-volatile alcohol-soluble matter of the shampoo. 72.41 g/100g is the noted value which lies within the required standards of IS 7884:2004. The result was reported in Table 5.

Table 5: The evaluation of non-volatile alcohol-soluble matter

Sample No.	Result	Unit
Herbal shampoo 1	73.75	gm/100 gm
Herbal shampoo 2	72.11	gm/100 gm
Herbal shampoo 3	71.37	gm/100 gm

e) Foam Height

The composition of activated carbon did not alter the foam height of the shampoo. 250 mm is the noted height which lies within the required standards of IS 7884:2004. The result was reported in Table 6 and Figure 1.

Table 6: The evaluation of foam height

Sample No.	Result	Unit
Herbal shampoo 1	250	mm
Herbal shampoo 2	250	mm
Herbal shampoo 3	250	mm



Figure 1: Foam Height

Foam Stability

In Water Base

The composition of activated carbon did not alter the foam stability of the shampoo. Thirty minutes was the noted time which lies within the required standards.

In Ethanol Base

Twenty-five minutes was the noted time. The foam which was stable in ethanol base can be used in soil remediation. Here the foam produced, irrespective of carbon content was stable on diluted ethanol base. So it could be used in the process of soil remediation.

Dirt Dispersion

The dirt dispersion ability increased as the concentration of the charcoal increased in the shampoo. It was shown in Figure 2.



Figure 2: Dirt Dispersion test

Microbiological Assay

The reduction count of microbes was found to be high, as the concentration of the activated charcoal in the shampoo was increased. The result was reported in Table 7.

Table 7: The evaluation of microbiological assay

Sample No.	Result	Unit
Herbal shampoo 1	900000	cfu/g
Herbal shampoo 2	600000	cfu/g
Herbal shampoo 3	200000	cfu/g

Critical Micelle Concentration

The CMC was studied and a graph was plotted. The result was shown in figure 3.

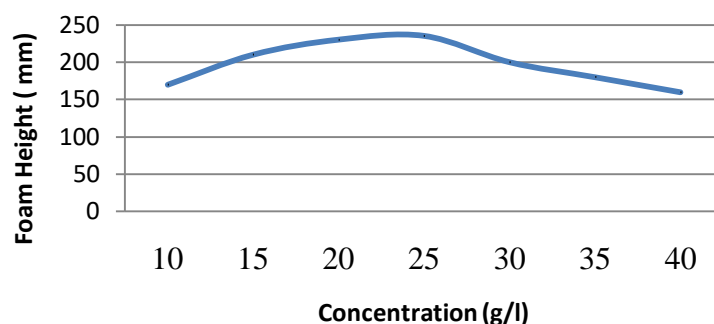


Figure 3: Foam height versus concentration

VI. CONCLUSION

Activated carbon was applied in various fields due to its adsorption and dirt removing capacity. In this paper usage of activated carbon in effectively removing dandruff and dirt is discussed. Based on the results obtained, it could be interpreted that usage of activated carbon in shampoo in various compositions yield better results in dandruff treatment and dirt dispersion. Since it was used along with herbs, the shampoo does not have any side effects.

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Chemistry Synthesis, Characterization and Photocatalysis of $\text{KSr}_2\text{Nb}_5\text{O}_{15}$ Doped with Nickel

By Paulo César Faria

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Abstract- Potassium and strontium niobate ($\text{KSr}_2\text{Nb}_5\text{O}_{15}$) of tungsten structure (TB) bronze was doped with nickel (Ni^{2+}) with stoichiometry type $\text{KSr}_2\text{Ni}_x\text{Nb}_{5-x}\text{O}_{15.6}$ onde $x = 0,25; 0,50$ and $0,75$. The powders were prepared by chemical synthesis using the modified Polyol method, and characterized by infrared spectroscopy with Fourier transform (FT-IR), by X-ray diffraction (XRD), including the network parameters (relative intensity - $I_{R(\text{KSNNi0,50})} > I_{R(\text{KSNNi0,25})} > I_{R(\text{KSNNi0,75})}$) and crystallite size. The effects of doping (Ni^{2+}) in the process of photo degradation were studied, the absorbance as a function of time for the Basic Blue dye 41 result from the photo degradation performed with the photo catalyst KSNNi0,50 presented a lower absorbance value, and the spectrophotometer was used in the UV-vis region. The efficiency and kinetics of better photo catalytic degradation of Basic Blue 41 by catalysts $\text{KSr}_2\text{Ni}_x\text{Nb}_{5-x}\text{O}_{15.6}$ where $x = 0,25; 0,50$ and $0,75$ was the system $\text{KSr}_2\text{Ni}_x\text{Nb}_{5-x}\text{O}_{15.6}$ where $x = 0,50$ which was discussed.

Keywords: *heterogeneous catalysis; oxidative catalysts; photo catalysts, niobate; tetragonal tungsten bronze structure; basic blue 41.*

GJRE-C Classification: For Code: 290601



CHEMISTRY SYNTHESIS CHARACTERIZATION AND PHOTOCATALYSIS OF $\text{KSr}_2\text{Nb}_5\text{O}_{15}$ DOPED WITH NICKEL

Strictly as per the compliance and regulations of:



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Chemistry Synthesis, Characterization and Photocatalysis of $\text{KSr}_2\text{Nb}_5\text{O}_{15}$ Doped with Nickel

Paulo César Faria

Abstract- Potassium and strontium niobate ($\text{KSr}_2\text{Nb}_5\text{O}_{15}$) of tungsten structure (TB) bronze was doped with nickel (Ni^{2+}) with stoichiometry type $\text{KSr}_2\text{Ni}_x\text{Nb}_{5-x}\text{O}_{15-\delta}$ onde $x = 0,25; 0,50$ and $0,75$. The powders were prepared by chemical synthesis using the modified Polyol method, and characterized by infrared spectroscopy with Fourier transform (FT-IR), by X-ray diffraction (XRD), including the network parameters (relative intensity - $I_{R(\text{KSNNiO},50)} > I_{R(\text{KSNNiO},25)} > I_{R(\text{KSNNiO},75)}$) and crystallite size. The effects of doping (Ni^{2+}) in the process of photo degradation were studied, the absorbance as a function of time for the Basic Blue dye 41 result from the photo degradation performed with the photo catalyst $\text{KSNNi}_{0,50}$ presented a lower absorbance value, and the spectrophotometer was used in the UV-vis region. The efficiency and kinetics of better photo catalytic degradation of Basic Blue 41 by catalysts $\text{KSr}_2\text{Ni}_x\text{Nb}_{5-x}\text{O}_{15-\delta}$ where $x = 0,25; 0,50$ and $0,75$ was the system $\text{KSr}_2\text{Ni}_x\text{Nb}_{5-x}\text{O}_{15-\delta}$ where $x = 0,50$ which was discussed.

Keywords: heterogeneous catalysis; oxidative catalysts; photo catalysts, niobate; tetragonal tungsten bronze structure; basic blue 41.

1. INTRODUCTION

The chemical contamination of the water covers a large spectrum of pollutants. The textile and leather industries are mainly responsible for the discharge of these large quantities of dyes. The highly colored effluents from these industries are affecting the nature of water, inhibiting the penetration of sunlight and reducing photosynthetic reactions, ATAR et al. (2008)^[1]. Approximately 10-15% of these dyes are not fixed to the substrates during the dyeing process, as some dyes used in the textile industry and their biodegradation by-products may have a high degree of toxicity, mutagenicity and carcinogenicity to humans; FRAGAA, ZANONIA (2009)^[2]. These Dye effluents are difficult to treat by conventional methods and cannot be completely degraded. Heterogeneous photo catalysis has aroused great interest, with the aim of efficiently purifying waste water containing dyes; ZHANG et al (2009)^[3].

Basic Blue 41 dye boasts an azo chromophore group and is used in the dyeing of synthetic fibers such as polyamide, polyester and viscose. Basic (cationic) dyes, which mostly have hydrolysis stability, are pH sensitive and are soluble in aqueous medium. Therefore, they require more efficient treatment methods

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for their complete removal in surface waters and effluents; FRAGAA, ZANONIA (2009)^[2].

The photo catalytic reactions on semiconductor surfaces are processed according to the basic steps of: excitation with light of energy greater than the band gap (E_g), of the semiconductor, generation of electron/gap pairs (e^-/h^+); imprisoning electrons and gaps by adsorbed species. The mechanism of redox processes mediated by semiconductors in aqueous media promotes the formation of the hydroxyl radical ($\text{OH}\cdot$), powerful oxidizing agent, generated by promoting the oxidation of the water / hydroxyl adsorbed by the lacuna, making the photo catalytic process highly efficient to oxidize most of the organic compounds. The efficiency of this process is related to the lifetime of the gaps ($h^+ + e^-$) and retardation of the recombination velocity of the generated loads (e^-/h^+) in the semiconductor; FRAGAA, ZANONIA (2009)^[2].

Although titanium dioxide is currently the most widely used, niobate based photo catalysts have been extensively studied because of their excellent photo catalytic properties, such as KNb_3O_8 , $\text{K}_6\text{Nb}_{10,8}\text{O}_{30}$, $\text{K}_4\text{Ce}_2\text{M}_{10}\text{O}_{30}$ ($M=\text{Ta}, \text{Nb}$), NiM_2O_6 ($M=\text{Nb}, \text{Ta}$), $\text{K}_4\text{Nb}_6\text{O}_{17}$, BiNbO_4 , NiO-KTiNbO_5 , etc. Among these niobate photo catalysts, $\text{K}_4\text{Ce}_2\text{M}_{10}\text{O}_{30}$ ($M=\text{Ta}, \text{Nb}$), NiM_2O_6 ($M=\text{Nb}, \text{Ta}$), NiO-KTiNbO_5 e $\text{K}_4\text{Nb}_6\text{O}_{17}$ high photo catalytic activity was found in the field of water decomposition. The KNb_3O_8 , $\text{K}_6\text{Nb}_{10,8}\text{O}_{30}$ and BiNbO_4 were studied for the degradation of dyes. However, the potassium and strontium type niobates (TTB) $\text{KSr}_2\text{Nb}_5\text{O}_{15}$ showed high photo catalytic activity in the degradation of red acid G under irradiation UV degrading and breaking the nitrogen double bond ($-\text{N}=\text{N}-$), and also causes the double bonds of the benzene and naphthalene rings to split up, the degradation rate exceeds 85% and its kinetics remains the first order; ZHANG et al. (2009)^[3].

Recent studies of the $\text{KSr}_2\text{Nb}_5\text{O}_{15}$ attests its photo catalytic activity resulting in methylene blue photo oxidation as a model reaction. These niobates $\text{KSr}_2\text{Nb}_5\text{O}_{15}$ present higher catalytic activity with lower calcination time, and under very low concentration conditions under irradiation UV; MATOS et al. (2017)^[4].

The present work aims to analyze the photo catalytic potential of materials with stoichiometry $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$, where its substitutions with nickel metal vary $0,25 \leq x \leq 0,75$, $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$.

$(\text{KSr}_2\text{Ni}_{0,25}\text{Nb}_{4,75}\text{O}_{15-\delta})$ - $(\text{KSr}_2\text{Ni}_{0,50}\text{Nb}_{4,50}\text{O}_{15-\delta})$ and $(\text{KSr}_2\text{Ni}_{0,75}\text{Nb}_{4,25}\text{O}_{15-\delta})$ calcined $1250^\circ\text{C}/1\text{h}$.

II. EXPERIMENTAL PROCEDURES AND CHARACTERIZATIONS

a) Synthesis

The Modified Polyol Method LI et al. (2006)[5], FELDMAN (2005)[6], Vivekanangan, Venkateswarlu, Satyanarayana (2004)[7], Xu, Huang, Long (2003)[8], Sun Et Al. (2002)[9]^[5-9], was used for the chemical synthesis of the following systems $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ (where $0,25 \leq x \leq 0,75$), $\text{KSr}_2\text{Ni}_{0,25}\text{Nb}_{4,75}\text{O}_{15-\delta}$; $\text{KSr}_2\text{Ni}_{0,50}\text{Nb}_{4,50}\text{O}_{15-\delta}$ e $\text{KSr}_2\text{Ni}_{0,75}\text{Nb}_{4,25}\text{O}_{15-\delta}$ Lanfredi Et Al. (2012)[10]; Dantas, Lanfredi, Nobre (2012)[11] E Gutiérrez, Saldivar, Lopez (2008)[12]^[10-12]. This method allows better control of reagents, low calcination temperatures, single-phase materials and high surface area dust, Lanfredi et al. (2014)[13]; Hsiao et al. (2012)[14]; LANFREDI et al. (2011)[15] e Nobre, Lanfredi (2003)[16]^[13-16].

The starting reagents for the synthesis by chemical method were nitric acid, HNO_3 (65,5% Reagen), strontium carbonate, SrCO_3 (99,0% Reagen), Potassium carbonate, K_2CO_3 (99,0% Reagen), ethylene glycol, $\text{HOCH}_2\text{CH}_2\text{OH}$ (98,0% Synth), nickel oxide, Ni_2O_3 (99,5% Reagen) and the ammoniacal oxalate salt of niobium, $\text{NH}_4\text{H}_2[\text{NbO}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ (CBMM-Brasil).

The precursor reagents for the preparation of the solution were dissolved in nitric acid with continuous stirring in the beaker. Then, 100 ml of ethylene glycol was added. The solution was heated to 90°C , promoting the decomposition of the NO_3 group, similar to the process developed in the Pechini method, NOBRE (1995)[17]; NOBRE (1999)[18]; PECHINI (1967)[19] e LANFREDI et al. (2014)[20]^[17-20]. After complete evaporation of the gas it was observed the formation of a polymeric gel (polyesterification reaction), where it was subjected to a pre-calcination. This polymer is maintained in the beaker to undergo a primary calcination in a box type furnace. This pre-calcination was performed in two stages. In the first step, from room temperature, the temperature was increased at a rate of $10^\circ\text{C}/\text{min}$ until it reaches the temperature of 150°C , which was held constant for 1 hour for the elimination of low molecular mass molecules, as water molecules. Following, maintaining the same heating rate, the temperature was raised to 300°C , being maintained at that temperature for 2 hours. This pre-calcination was carried out under a $\text{N}_2(\text{g})$ with flow of $500\text{ mL}/\text{min}$. The cooling was carried out at the natural rate of the furnace, maintaining the nitrogen flow.

The process results in partial decomposition of the polymer to form a resin, which consists of a brittle reticulated material. This material was deagglomerated

in agate-mortar and sieved in mesh 350 meshes with opening of $45\ \mu\text{m}$, being called precursor powder. Afterwards the steps of deagglomeration and sifting exhibiting fine and homogeneous show. Calcination was carried out at 1250°C during 1 h, heating rate of $10^\circ\text{C}/\text{min}$.

b) Characterization Techniques

The post-ceramics based on strontium and potassium niobate ($\text{KSr}_2\text{Nb}_5\text{O}_{15}$) with stoichiometry $\text{KSr}_2\text{Ni}_{0,25}\text{Nb}_{4,75}\text{O}_{15-\delta}$; $\text{KSr}_2\text{Ni}_{0,50}\text{Nb}_{4,50}\text{O}_{15-\delta}$ and $\text{KSr}_2\text{Ni}_{0,75}\text{Nb}_{4,25}\text{O}_{15-\delta}$ were characterized by absorption spectroscopy in the infrared region (FT-IR), X-ray diffraction (DRX) then its photocatalytic potential was evaluated.

- i. *Microscopia Eletrônica de Varredura (MEV)*
- ii. *Infrared with Fourier Transform (FT-IR)*

The infrared spectral absorption spectroscopy was performed in a spectrophotometer of the brand SHIMADZU model IRAffinity-1. The spectral range used was in the medium infrared region ($3500 - 450\text{ cm}^{-1}$), with resolution of 8 cm^{-1} and 120 scans. The samples were dispersed in KBr in the proportion of 1:100. Potassium Bromide tablets (KBr) were prepared using a tablet of the mark PIKE de 13 mm diameters in hydraulic press of 10 ton. Each tablet was produced by macerating $1,5\text{ mg}$ of the sample, ceramic powders, with $0,30\text{ g}$ de KBr in agate mortar. The mixture with KBr was pressed in the pellets by 10 min. The tablet thus obtained was analyzed in a spectrophotometer.

- iii. *X-ray diffractometry (XRD)*

The precursor post-ceramics, the solid solutions of $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$, where the $x = 0,250; 0,500$ e $0,750$ producing stoichiometrically the following materials: $\text{KSr}_2\text{Ni}_{0,25}\text{Nb}_{4,75}\text{O}_{15-\delta}$, $\text{KSr}_2\text{Ni}_{0,50}\text{Nb}_{4,50}\text{O}_{15-\delta}$ and $\text{KSr}_2\text{Ni}_{0,75}\text{Nb}_{4,25}\text{O}_{15-\delta}$ and treated at temperatures of 1250°C , were characterized by X-ray diffraction using a diffractometer SHIMADZU (model XRD-6000), with radiation $\text{Cu K } \alpha$ ($\lambda = 1,54060\ \text{Å}$), operating at 40 kV and 30 mA , in the range of $5 \leq 2\theta \leq 80$, with scan time of $1,00^\circ/\text{min}$, steps of $0,02^\circ$ and time per step equal to $1,20\text{ s}$. The slits of divergence and scattering used were $1,00^\circ$ and the receiving slot of $0,30\text{ mm}$.

- a. *Network Parameters*

The network parameters are calculated using the "Least Squares". The positions $2\theta_0$, the plans hkl, the type of the crystalline structure, the number of interactions to be executed, are the program data.

- b. *Average crystallite size*

The values of the mean crystallite size for the solid solutions of $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$, where a) $x = 0,25$; b) $x = 0,50$ and c) $x = 0,75$ were calculated by the Scherrer equation, equation (1).

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

being β the width of the peak where the intensity is half of its maximum value denominated (peak width at half height), θ is the angle corresponding to the diffraction, λ is the wavelength of the Cu ($1,5406 \text{ \AA}$), k is the constant of proportionality, called the particle shape factor ($TB = 0,89$) NUFFIELD (1986)^[21].

c) Evaluation of photo catalytic potential

For the study of photo catalytic activities, we used the degradation material Basic Blue 41 in aqueous solutions. The photo catalytic tests will be performed in order to maximize the activation of the post-ceramics as a catalyst.

d) Photo catalytic procedure of Basic Blue 41 dye

To investigate the photo catalytic activity of the ceramic powders, a photo catalytic reactor. This low power photo catalytic reactor has a source of irradiation, a light bulb with dimensions (length 438,0 mm, diameter 26,0 mm) HSN® 15 W G13 which emits radiation at the wavelengths UVC between 200 to 280 nm, that is, specifically 250 nm, which corresponds to the range of the electromagnetic spectrum of ultraviolet C (rays UV-C). In this way the photo catalytic activity of the material was studied using as comparative parameter tests with UV-C. For the light bulb the measured irradiance was 0,3 mW/cm² during the photo catalytic test stage and the measured irradiance of 1,8 mW/cm².

Basic blue 41 solutions were prepared by means of a dilution of the stock solution, thus obtaining a concentration of 40 ppm. The pH of the solution was then measured through a pH meter (GEHAKA) adjusting the pH to 8 in some tests with the aid of a few drops of sodium hydroxide 0,01 mol.L⁻¹ and in other tests the natural pH of the solution was maintained for the purpose of comparison with the original methodology using the ceramic powders. Was heavy 0,125g of catalyst, in order to obtain optimum concentration of catalyst (0,55 g.L⁻¹) in a volume of 1000 mL of solution (12,5 mg/L).

The first step consists of the photo catalytic tests of a period of 1 hour, where at the beginning of the first 60 minutes the post-ceramics were added in solution of Basic Blue 41, with the flow of water circulating through the reactor, with the help of a small compressor, in order to reach the equilibrium of adsorption-desorption in the dark, this is the step in the dark, process of accommodation of the material to solution. The last 30 minutes with the reactor lamp on and continuous flow. In the last minutes (photolysis) at 59 minutes, a 5.0 mL aliquot of the sample was collected by means of a volumetric pipette, starting the removal of the first sample, identified (t = 0).

The second step of the photo catalytic tests were the collections every 15 minutes totaling 1 h, and identified= 1, 2, 3 e 4.

The third step of the photo catalytic tests, the collections were performed every 30 minutes, totaling 2 h and identified by t= 5, 6, 7, 8, 9 e 10, totaling 11 samples collected.

In the fourth step, the collected samples were accommodated in test tubes enclosed in boxes avoiding exposure to light. These test tubes were centrifuged, 3000 rpm by 20 minutes, in order to separate the catalyst from the solution, 0,5 mL of the sample (supernatant) with the aid of a Pasteur pipette, and diluted 5 mL of distilled and deionized water, measured with a volumetric.

In the fifth step, the supernatants were analyzed in a UV-vis spectrophotometer and programmed for wavelength reading (λ) 611 nm of the solution, recording the absorbance of the samples.

e) Determinação da Eficiência e Parâmetro Cinético de Degradação

The photo catalytic activity of post-ceramics $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$, where $x = 0,25; 0,50$ and $0,75$ the kinetics of the disappearance of Basic Blue 41 as test molecule.

In order to establish the photo catalytic behavior of niobate based materials, both degradation kinetics and direct photolysis in the absence of solids were followed. The disappearance of the Basic Blue dye 41 was reported in terms of the conversion (X) obtained by the following expression of the efficiency represented by the concentration equation (2):

$$X = \left[\left(\frac{C_0 - C_t}{C_0} \right) \right] \cdot 100 \cong \left[\frac{(A_0 - A_t)}{A_0} \right] \cdot 100 \quad (2)$$

Where C_0 is the initial concentration of Basic Blue dye 41, C_t is the concentration in reaction time t , and a_0 and a_t are the initial absorbance and the absorbance at the reaction time t . In this way he analyzed and studied the efficiency and kinetics of degradation of the dye Basic Blue 41.

III. RESULTS AND DISCUSSIONS

a) Infrared with Fourier Transform (FT-IR)

Figure 1 (a, b e c) - ($\text{KSr}_2\text{Ni}_{0,25}$; $\text{KSr}_2\text{Ni}_{0,50}$ and $\text{KSr}_2\text{Ni}_{0,75}$) shows the infrared (IR) vibration absorption spectra for the stoichiometric systems $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ onde a) $x = 0,25$; b) $x = 0,50$ and c) $x = 0,75$. These spectra present bands in the range of 450 to 1100 cm^{-1} , such bands are characteristic of the connections Nb-O for regions of low frequencies, attributed to the antisymmetrical stretching for the connection $\nu_{as}(\text{Nb-O})$, symmetrical stretching $\nu_s(\text{Nb-O-Nb})$, symmetrical stretching $\nu_s(\text{Nb-Oap})$. The displacements in the spectra show that the bonds between niobium and oxygen cations are shorter in relation to host structure ($\text{KSr}_2\text{Nb}_5\text{O}_{15}$) DENIO et al. (2010)[22]; YEBIN, GUOHUA, HUA (2003)[23] and

BERGAMASCHI (2000)[24][22-24], this system has characteristic bands that identify the presence of an "envelope" and with the addition of nickel (Ni-O) to $\text{KSr}_2\text{Nb}_5\text{O}_{15}$; YEBIN, GUOHUA, HUA (2003)[23] tends to

displace the system bands. Symmetrical stretching is attributed (ν_s) and antisym metric (ν_{as}) DENIO et al. (2010)[22] and BERGAMASCHI (2000)[24].

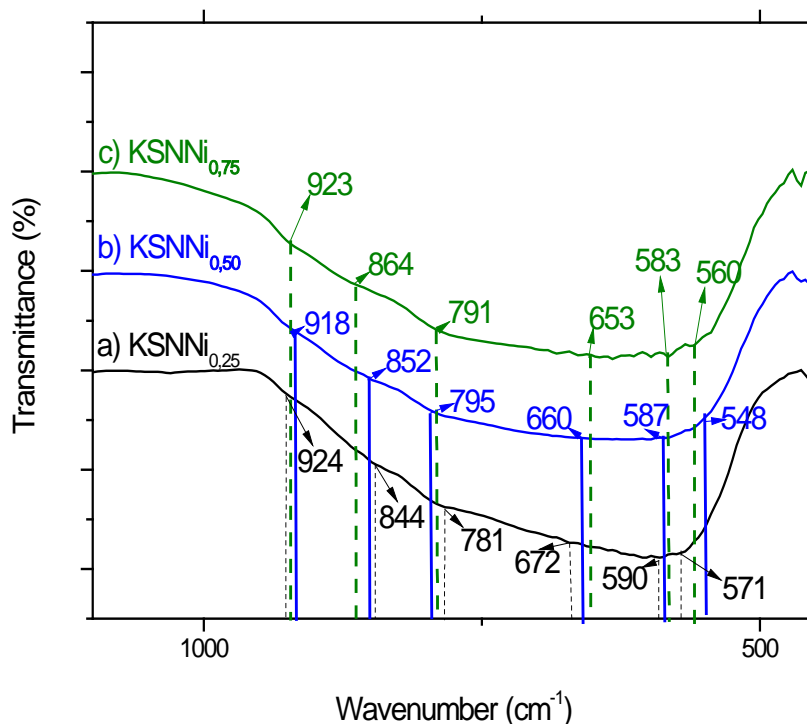


Fig. 1: Vibrational spectrum in the region of $450\text{--}1100\text{ cm}^{-1}$ of infrared, for stoichiometric systems $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where a) $x = 0,25$; b) $x = 0,50$ and c) $x = 0,75$ denominated $\text{KSNNi}_{0,25}$, $\text{KSNNi}_{0,50}$ and $\text{KSNNi}_{0,75}$.

Figure 1 (a) - ($\text{KSNNi}_{0,25}$) shows a portion of the spectrum in the infrared region where it is characterized by an envelope in the region between $450\text{--}1100\text{ cm}^{-1}$ for the system $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where a) $x = 0,25$. In this figure, the spectra show bands of strong intensity below 1000 cm^{-1} , characteristic of niobates. The bands identified have wavelengths in $571, 590, 672, 781, 844$ and 924 cm^{-1} such band refer to the oxide-metal bonds.

Figure 1 (b) - ($\text{KSNNi}_{0,50}$) shows parts of the spectrum in the infrared region where it is characterized by an envelope in the region between $450\text{--}1100\text{ cm}^{-1}$ for the system $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where b) $x = 0,50$. In this figure, the spectra show bands of moderate intensity below 1000 cm^{-1} , characteristic of niobates. The bands identified have wavelengths in $548, 587, 660, 795, 852$ and 918 cm^{-1} such band are attributed to the oxide-metal bonds.

Figure 1 (c) - ($\text{KSNNi}_{0,75}$) shows parts of the spectrum in the infrared region where it is characterized by an envelope in the region between $450\text{--}1100\text{ cm}^{-1}$ for the system $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where c) $x = 0,75$. In this figure, the spectra show bands of moderate intensity below 1000 cm^{-1} , characteristic of niobates.

The bands identified have wavelengths in $560, 583, 653, 791, 864$ and 923 cm^{-1} such band are attributed to the oxide-metal bonds. Table 1 below shows the low intensity and high intensity wave numbers for stoichiometric systems $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,25; 0,50$ and $0,75$.

Table 1: Absorption band positions, associated with each stoichiometry of the $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0; 0,25; 0,50$ and $0,75$.

Stoichiometric system	Wavenumber (cm^{-1})					
$\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$	571	590	672	781	844	924
$\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$	548	587	660	795	852	918
$\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$	560	583	653	791	864	923

From the data in Table 1 it was observed a displacement of the bands characteristic of the connections Nb-O and Nb-O-Nb for regions of low frequencies with increasing concentration of nickel cations. The narrower and lower intensity bands move to regions with higher wave numbers, around 571 to 590 cm^{-1} to $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$; 548 to 587 cm^{-1} to $\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$ and 560 to 583 to $\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$. These bands are attributed to stretching (Nb-O). The medium and wide asymmetric bands in the regions of 672 cm^{-1} to 781 cm^{-1} , observed in the spectrum of $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$ they move to regions between 660 cm^{-1} to 795 cm^{-1} spectra of the solid solutions of $\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$ and this system for 653 cm^{-1} to 791 cm^{-1} for the system $\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$. The bands in the region between 844 cm^{-1} to 924 cm^{-1} can be attributed to the symmetrical (Nb-O-Nb) LANFREDI, FOLGUERAS-DOMÍNGUES, RODRIGUES (1995)^[25]. The displacement of these bands to the region of smaller wave number in the spectra of the solid solutions of the $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$, $\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$ and $\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$, shows that the bonds between niobium and oxygen cations are shorter.

b) X-ray diffractometry (XRD)

Figure 2 shows the X-ray diffractograms obtained for the precursor powders of the stoichiometric system based on niobate $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,25; 0,5$ and $0,75$ heat treated at 1250°C for 1 hour, in an oxygen atmosphere. X-ray diffractograms show an increase in the definition of diffraction peaks to 1250°C , associated with the decrease of the microdeformation of the net and increase of the structural stability. According, LANFREDI et al. (2005)^[26], in solid solutions of $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ is adopted the valence of the Ni^{2+} , since the oxidation state +3 of nickel cation (Ni) has been rarely detected. The substitution of radium cations (r) such as the Sr^{2+} ($r_{\text{Sr}^{2+}} = 1,18 \text{ \AA}$) by cations of lightning (r) small, as the Ni^{2+} ($r_{\text{Ni}^{2+}} = 0,69 \text{ \AA}$) not favorable. In addition, the cations of Ni^{2+} show strong preference for octahedral coordination, the same coordination of niobium (Nb). In this sense, the ionic radius of Nb cations in a high oxidation state ($r_{\text{Nb}^{5+}} = 0,64 \text{ \AA}$), similarity with the ionic radius of the Ni^{2+} . However, the valence difference is equal to three units, which is not favorable. However, the best similarity of the ionic Ni^{2+} occurs for the Nb^{5+} partially reduced to Nb^{4+} ($r_{\text{Nb}^{4+}} = 0,68 \text{ \AA}$). Here it is important to comment that the Nb^{4+} represents a partial reduction of the niobium

cation, whereas a completely reduced state is given by the niobium with valence 3+, Nb^{3+} , ($r_{\text{Nb}^{3+}} = 0,72 \text{ \AA}$).

The structural characterization of the post-ceramics constituted by the $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,25; 0,50$ e $0,75$ obtained by the modified Polyol chemical method was investigated and analyzed by means of the X-ray diffraction technique. The standard XRD for the system $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ with a range of 2θ of $5^\circ - 80^\circ$ is shown in Fig. 1 (0,25; 0,50 e 0,75). According to the crystal data file JCPDS 34-0108 (2000)^[27], the system is of tetragonal type and presents spatial group P4bm (100). The diffractograms of the samples that were prepared and calcined at $1250^\circ \text{C} / 1\text{h}$ fit the pattern, and in some angles (θ) present significantly lower displacements, which may consider that these samples consist of single phase for the compounds with variation of doping. The Diffractogram shown in figure 2 (0,25) referring to the system $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$ where $x = 0,25$ presents formation of a single crystalline phase based on the tetragonal symmetry of the crystallographic sheet JCPDS: 34-0108 (2000)^[27] related to the niobium oxide strontium and potassium ($\text{KSr}_2\text{Nb}_5\text{O}_{15}$). as for the values of the müeller index (hkl), of 2θ , of θ , of the interplanar distances (nm) and the intensities can be seen in table 1 below.

Figure 2 (0.50) shows the X-ray diffractogram obtained by the system precursor powder $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x=0,50$ a $1250^\circ \text{C}/1\text{h}$ calcined in an oxygen atmosphere. The diffractogram, similar to figure 1 (0.25), shows crystalline phase formation indicated in the indexing of the crystallographic data sheet JCPDS: 34-0108 (2000)^[27] tetragonal symmetry for this system $\text{KSr}_2\text{Nb}_5\text{O}_{15}$ the narrow peaks indicating an increase in the crystallinity of the calcined material are observed in this diffractogram (Fig. 2 (0.25, 0.50 and 0.75)) the $1250^\circ \text{C} / 1\text{h}$.

Figure 2 (0.75) shows the X-ray diffractogram obtained by the $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x=0,75$ calcined $1250^\circ \text{C} / 1\text{h}$ and obtained in an oxygen atmosphere. This characterization showed the formation of the monophasic and crystalline powder ($\text{KSr}_2\text{Ni}_{0,75}$). According to the data of this record, the phases found have a tetragonal structure compatible with the spatial group P4bm (100).

i. Network Parameters

The values given in tables 2 and 3 refer to the crystallographic sheet JCPDS: 34-0108 (2000)^[27] shows the network parameters used to obtain the results of the interplanar distances and the θ .

Table 2: Values of müeller index (hkl), of 2θ , of θ , of interplanar distances (nm) and the intensities, acquired in the crystallographic sheet JCPDS: 34-0108 (2000)[27] for the system $\text{KSr}_2\text{Nb}_5\text{O}_{15}$.

Hkl	2θ	θ	$d_{\text{interplanar}}$ (nm)	Intensidade
(400)	28.626	14.313	3.1	12
(311)	32.076	16.038	2.8	100
(002)	46.013	23.01	1.9	36
(422)	57.205	28.6025	1.6	31
(731)	61.297	30.65	1.5	6
(622)	67.112	33.56	1.4	7

However, the results data referring to table 3 refers to stoichio metric systems $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$. $\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$. $\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$ doped with nickel, where $x = 0,25; 0,50$ e $0,75$.

Table 3: Values of the müeller index (hkl), of 2θ , of θ , of interplanar distances (nm) and the intensities for stoichio metric systems $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$. $\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$. $\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$.

Hkl	2θ	θ	$d_{\text{interplanar}}$ (nm)	Intensidade
$\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$	27,9	13,95	3,2	1.003,5
	29,7	14,85	3,0	1.300,3
	32,3	16,15	2,8	2.308,9
$\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$	27,9	13,95	3,2	2.449,4
	29,7	14,85	3,0	3.047,3
	32,2	16,1	2,7	5.104,6
$\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$	27,9	13,95	3,2	1.631,8
	29,8	14,9	2,9	1.865,1
	32,2	16,1	2,7	3.455,5

The relative intensity (IR) for stoichio metric systems $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$, $\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$ e $\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$ follows the descending order.

$$I_{R(\text{KSNNO},50)} > I_{R(\text{KSNNO},25)} > I_{R(\text{KSNNO},75)}$$

ii. Crystallite size

The crystallite size of the solid solutions $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,25; 0,5$ and $0,75$ was determined using the program Jade 8 Plus. The widening of the mean width at half height (FWHM) of the diffraction peaks of the experimental diffractogram was considered. The Jade 8 Plus program calculates the

mean crystallite size by applying the Scherrer equation; AZAROFF, BUERGUER (1958)^[28]. Where D is the average crystallite size, k is the proportionality constant, which depends on the shape of the particles ($TB = 0,89$)², λ is the wavelength of the Cu ($1,5406 \text{ \AA}$), β is the width at half height of the corrected peak and θ the angle corresponding to the diffraction. The instrumental factors were corrected using the Silicon (Si) standard. The crystallite size KSN pure and doped with Nickel Method Modified polyol calcined for 1 hour, follows table 4.

Table 4: Mean crystallite size for solid solutions of stoichiometric systems $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$; $\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$ $\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$ doped with nickel where $x = 0,25; 0,50$ e $0,75$ and calculated by the Scherrer equation.

Stoichiometric system	Crystallite size - calcination at 1250 °C / 1h
$\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$	20,85 nm
$\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$	23,70 nm
$\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$	27,00 nm

The largest crystallite size was observed for solid solution $\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$. The increase in the mean crystallite size with increasing the value of x to 0.75 is related to a greater distortion of the unit cell, increasing the diffusion process and nucleation of the crystals; MELO (2007)^[29]. These values show that the increased dopant concentration promotes an increase in structural anisotropy in the material; DANTAS et al.

(2009)^[30]. According to the values in Table 4, the value of the average size of network crystallite increases as the x value increases from 0.25 to 0.75. This effect is a consequence of the high degree of doping of the host structure, where an excess of nickel cations causes a disorder in the crystalline lattice to form defects, caused by non-stoichio metry of the structure; WANG et al. (2012)^[31].

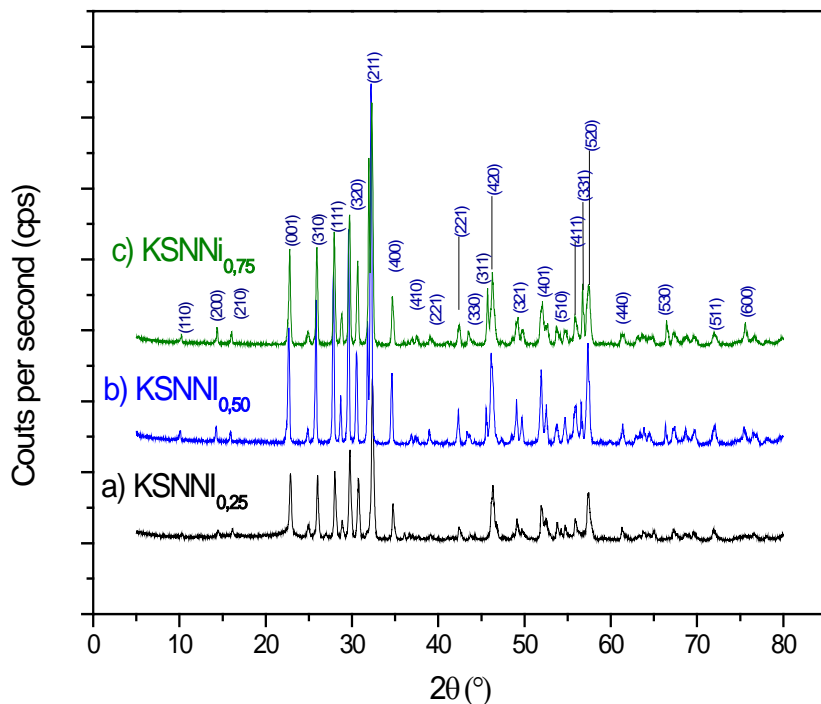


Fig. 2: Difractograma de raios X dos sistemas estequiométricos $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$ – ($\text{KSNi}_{0,25}$), do $\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$ – ($\text{KSNi}_{0,50}$) e do $\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$ – ($\text{KSNi}_{0,75}$) calcinados a $1250\text{ }^\circ\text{C}$ / 1h em atmosfera de oxigênio.

In this diffractogram between 20 and 35 displayed on the 2θ scale there is a close junction between peaks in relation to figure 2, the crystal plug, JCPDS: 34-0108 (2000)^[27] follows partially offset from the peaks. The values of the interplanar distances, the relative intensity and the 2θ of the diffractogram coincide with the values listed on this sheet. The lines of this chart coincide with the diffractogram peaks, however, it is possible to notice a singular difference between these peaks, as regards the relative intensity with the lowest intensity (Figure 2 - c) $\text{KSNi}_{0,75}$ in relation to the relative intensity of b) $\text{KSNi}_{0,50}$ in Figure 2, where in this diffractogram the intensity of the peaks is much more expressive and defined, but of a considerable narrow width.

c) *Avaliação do potencial fotocatalítico: Determinação da eficiência e Cinética de degradação*

Figure 3 - a) $\text{KSNi}_{0,25}$; b) $\text{KSNi}_{0,50}$ and c) $\text{KSNi}_{0,75}$ shows the absorbance as a function of time, the adsorption in the dark for 1h and the degradation rate with the reactor connected for 3h together with the photo degradation kinetics with irradiation of UV light. Figure 4 shows the rate of discoloration over time for a) $\text{KSNi}_{0,25}$; b) $\text{KSNi}_{0,50}$ and c) $\text{KSNi}_{0,75}$ and Fig. 5 shows the $\ln(C_0/C_t)$ depending on the time for the systems (a) $\text{KSNi}_{0,25}$; b) $\text{KSNi}_{0,50}$ and c) $\text{KSNi}_{0,75}$ to investigate the degradation of Basic Blue 41. The performance of the materials synthesized here can be

observed that the rate of adsorption is very fast for all catalysts, reaching the equilibrium of the dye after 30-60 min. Thus, photocatalytic tests with UV light irradiation were performed after an initial 60 min adsorption period. It is interesting to note that the amount of Basic Blue 41 adsorbed has decreased over time.

d) *Efficiency and rate of degradation of $\text{KSNi}_{0,25}$; $\text{KSNi}_{0,50}$ and $\text{KSNi}_{0,75}$*

The first test was performed with the stoichiometry material $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,25$ irradiated with UV light and catalyst 0,10g and concentration of $12,5\text{ mg}\cdot\text{L}^{-1}$ of the type dye (Basic Blue 41). Figure 3 shows the absorbance versus time of Basic Blue 41 using the material $\text{KSNi}_{0,25}$. The moment the material is added $\text{KSNi}_{0,25}$ the elapsed time solution of 1 h without UV irradiation at a drop in absorbance of 2.35%, This percentage is due to the fact of an accommodation of the solution to the surface of the catalyst, being that at the moment of the adsorption there was no degradation of the solution of Basic Blue 41. However, for the same material $\text{KSNi}_{0,25}$ considering the last three hours the rate of discoloration was 91,35 % considering its absorbance, which leads us to believe that the degradation of Basic Blue 41 can be attributed to the photo catalytic effect of $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,25$ under irradiation UV.

The second test performed with stoichiometry material $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,50$ irradiated with UV light and catalyst 0,10g and concentration of $12,5 \text{ mg.L}^{-1}$ of the dye (Basic Blue 41). Figure 3 shows the absorbance versus time of Basic Blue 41 using the material Figure 3 shows the absorbance versus time of Basic Blue 41 using the material $\text{KSNNi}_{0,50}$. The moment the material is added $\text{KSNNi}_{0,50}$ the elapsed time of 1 h, without UV irradiation at a drop in absorbance of 4,00 %. This percentage is due to the fact that a solution solution to the surface of the catalyst, indicating a larger surface, and that at the moment of adsorption there was no degradation of the solution of Basic Blue 41. However, for the same material $\text{KSNNi}_{0,50}$ considering the last three hours the rate of discoloration was 97,51 % considering its absorbance, which leads us to believe that the degradation of Basic Blue 41 can be attributed to the photo catalytic effect of $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,50$ under irradiation UV.

The third test performed with the stoichiometry material $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,75$ irradiated with UV light and catalyst 0,10g and concentration of $12,5 \text{ mg.L}^{-1}$ of the dye (Basic Blue 41). Figure 3 shows the absorbance versus time of Basic Blue 41 using the material $\text{KSNNi}_{0,75}$. The moment the material is added $\text{KSNNi}_{0,75}$ the elapsed time of 1 h, without UV irradiation at a drop in absorbance of 3,00 %, This percentage is

due to the fact that a solution to the surface of the catalyst, indicating a larger surface, and that at the moment of adsorption there was no degradation of the solution of Basic Blue 41. However, for the same material $\text{KSNNi}_{0,75}$ considering the last three hours the rate of discoloration was 97,51 % considering its absorbance, which leads us to believe that the degradation of Basic Blue 41 can be attributed to the photo catalytic $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,75$ under irradiation UV.

It is important to emphasize that the adsorbed samples presented expected results due to the chemical nature of Basic Blue 41, this dye being a strong Lewis base, and its adsorption is thermodynamically favored by Lewis acidic solids, such as the materials synthesized here. According to surveys; MATOS et al. (2017)^[4], in fact, these materials that are Lewis acids constituted of niobates present high electronic affinity of their ions Nb (86,1 kJ / mol). This condition induces the agreement that niobate based catalysts have a more acidic surface pH and therefore have a high affinity for basic amines such as Basic Blue 41 with a high dissociation constant (pKb) and a high neutralization potential.

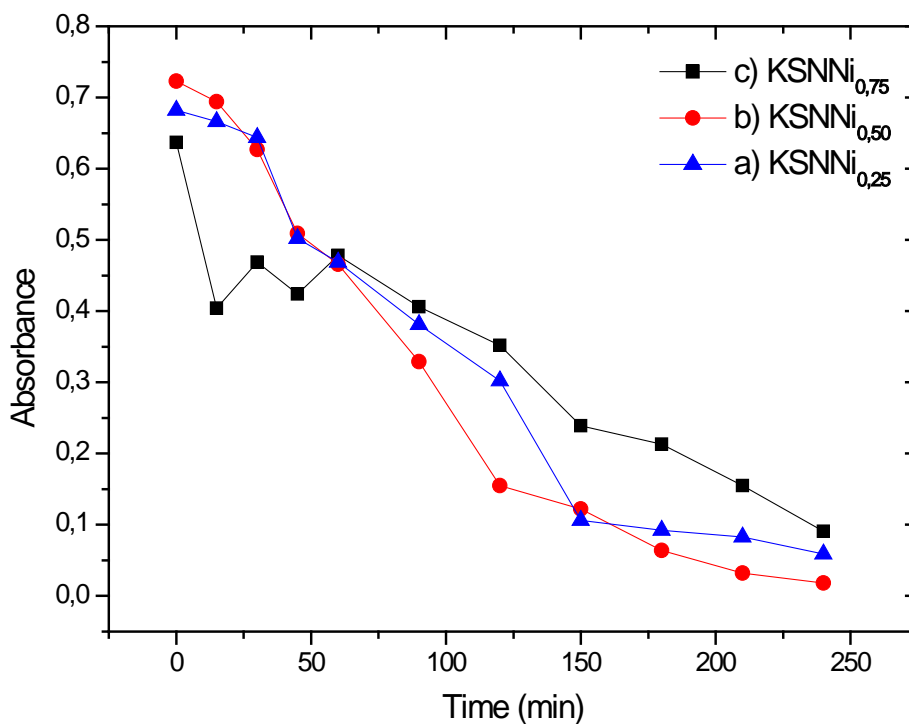


Fig. 3: Absorbance as a function of the degradation time of the Basic Blue 41 using the materials a) $\text{KSNNi}_{0,25}$; b) $\text{KSNNi}_{0,50}$ and c) $\text{KSNNi}_{0,75}$.

Absorbance readings at the maximum absorption wavelength of the "basic blue 41" dye were performed for the photo catalytic tests with the photo catalysts $\text{KSNNi}_{0,75}$; $\text{KSNNi}_{0,50}$ and $\text{KSNNi}_{0,25}$. According to figure 3 - a) $\text{KSNNi}_{0,25}$; b) $\text{KSNNi}_{0,50}$ and c) $\text{KSNNi}_{0,75}$ a decrease in the absorbance values versus time for all tests was observed indicating a decrease in the concentration of the basic blue 41 dye in solution resulting from the photo degradation. Among the photo

catalytic tests performed, the photo catalyst test $\text{KSNNi}_{0,50}$ presented lower absorbance value.

From Figure 4 - a) $\text{KSNNi}_{0,25}$; b) $\text{KSNNi}_{0,50}$ and c) $\text{KSNNi}_{0,75}$ an increase in the rate of decolorization as a function of time is observed for all the photo catalytic tests performed, indicating a decrease in the concentration of the dye "basic blue 41". The photo catalyst that presented the highest discoloration rate was the $\text{KSNNi}_{0,50}$ so it was the most efficient.

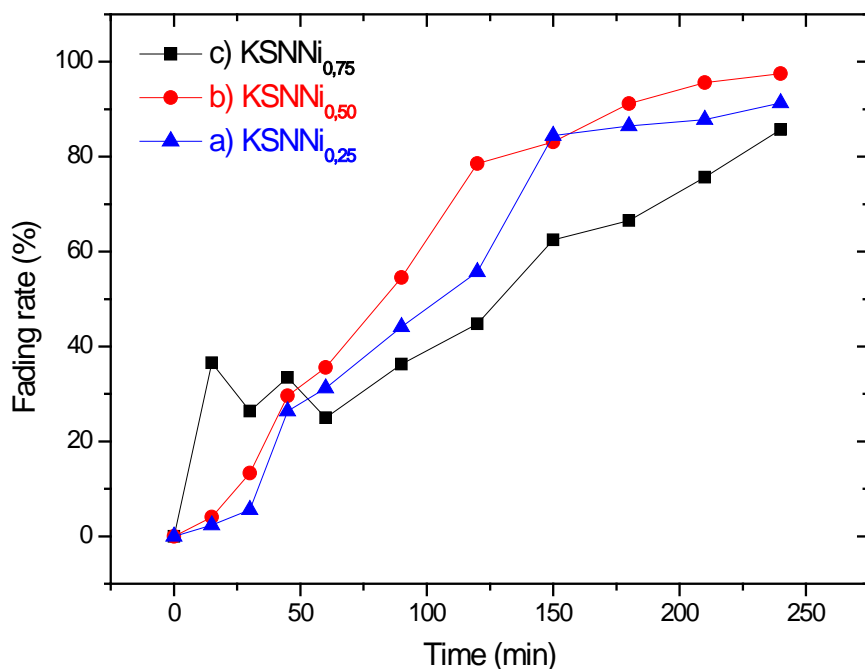


Fig. 4: Discoloration rate versus time for systems a) $\text{KSNNi}_{0,25}$; b) $\text{KSNNi}_{0,50}$ and c) $\text{KSNNi}_{0,75}$.

e) Kinetics of degradation of materials $\text{KSNNi}_{0,25}$; $\text{KSNNi}_{0,50}$ and $\text{KSNNi}_{0,75}$

Figure 5 - a) $\text{KSNNi}_{0,25}$; b) $\text{KSNNi}_{0,50}$ and c) $\text{KSNNi}_{0,75}$ shows the linear relationship $\ln(C_0 / C_t)$ as a function of the photo degradation reaction time t which indicates that the photo degradation reaction of the catalysts continues to be first order kinetics. For the catalyst $\text{KSr}_2(\text{Ni}_{0,25}\text{Nb}_{4,75})\text{O}_{15-\delta}$ which shows the linear relationship $\ln(C_0 / C_t)$ and photo degradation reaction time t indicates that the reaction continues being kinetic of first, according to kinetic equation. For Fig. 5 - b) $\text{KSNNi}_{0,50}$ shows the linear relationship $\ln(C_0 / C_t)$ and photo degradation reaction time t indicates that the photo degradation reaction of the catalyst $\text{KSr}_2(\text{Ni}_{0,50}\text{Nb}_{4,50})\text{O}_{15-\delta}$ also follows kinetics of first order, according to kinetic equation. For fig.5 - c) $\text{KSNNi}_{0,75}$ shows the linear relationship $\ln(C_0 / C_t)$ and photo degradation reaction time t indicates that the photo degradation reaction of the catalyst $\text{KSr}_2(\text{Ni}_{0,75}\text{Nb}_{4,25})\text{O}_{15-\delta}$ also follows first-order kinetics, according to kinetic equations.

On the other hand, the Figure 3 - a) $\text{KSNNi}_{0,25}$; b) $\text{KSNNi}_{0,50}$ and c) $\text{KSNNi}_{0,75}$ show very similar kinetic trends in the photo catalytic degradation of Basic Blue 41 for all photo catalysts $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,25; 0,5$ and $0,75$. To compare the photo activity, the first order apparent rate constants (k_{app}) were estimated from linear regression of kinetic data, and assuming that Basic Blue 41 photo degradation follows a first order reaction rate mechanism; FRAGAA, ZANONIA (2009)[2] e ATAR, OLGUN, ÇOLLAK (2008)[1] ^[1-2]. We attribute this photo activity due to the presence of apical oxygen atoms that are very reactive and are attached to the niobium in the octahedron $[\text{NbO}_6]$ MATOS et al. (2017)^[4].

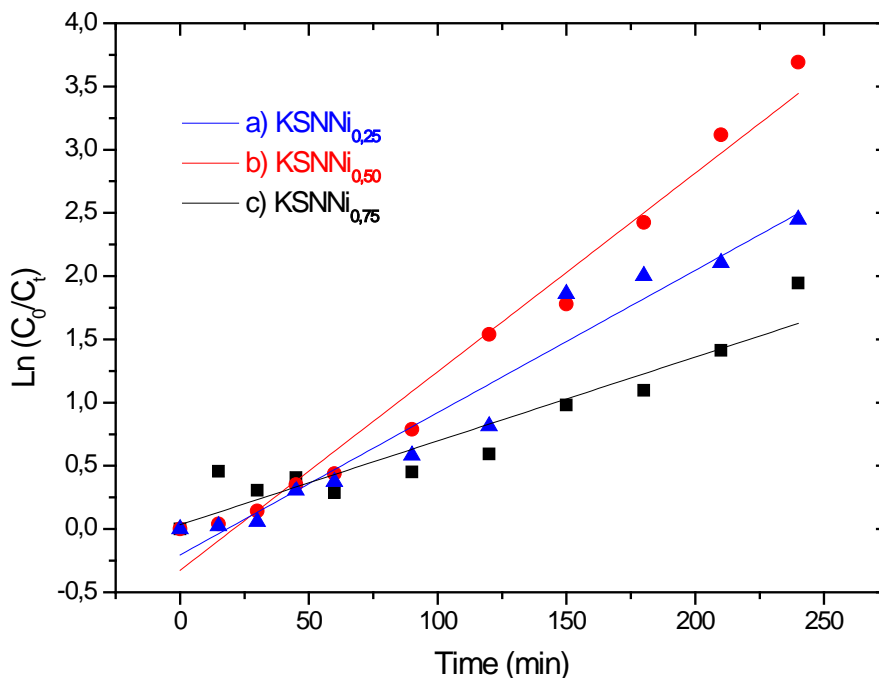


Fig. 5: $\text{Ln}(C_0/C_t)$ depending on the time for the systems a) $\text{KSNNi}_{0,25}$; b) $\text{KSNNi}_{0,50}$ and c) $\text{KSNNi}_{0,75}$.

Analysis of the kinetic curves in all the photo catalytic tests performed shows a first order kinetics, the data shown in Table 1 confirm such observations.

Table 1: Summary of the kinetic parameters for the photocatalytic tests

Catalyzer	$\text{KSNNi}_{0,25}$	$\text{KSNNi}_{0,50}$	$\text{KSNNi}_{0,75}$
K (min)	0,01125	0,01571	0,00663
$t_{1/2}$ (min)	61,61	44,12	104,55
R^2	0,95017	0,97412	0,88381
Kinetic Equation	$\text{Ln}(C_0/C_t) = -0,20445 + 0,01125x$	$\text{Ln}(C_0/C_t) = -0,32642 + 0,0151x$	$\text{Ln}(C_0/C_t) = 0,03394 + 0,00663x$

The photo catalytic test with the photo catalyst $\text{KSNNi}_{0,5}$ presented a higher speed constant and a shorter half-life, being the most efficient. According to the results, the nickel doping in the host structure of the $\text{KSr}_2\text{Nb}_5\text{O}_{15}$ promotes an increase of the photo catalytic activity until a substitution of 10% in mol of Ni^{2+} , and a decrease in photo catalytic activity can be observed for the KSN doped with 15 mol%.

IV. GENERAL DISCUSSIONS

Solid state diffusion is a mass transport medium occurring within the solid materials according to an atomic movement occurring in stages. The mechanisms promoting this mass transport are realized by means of an exchange of atoms which is located in a normal position of the network with an adjacent gap or by a migration from an interstitial position to an adjacent empty interstitial position in which the host metal, the interstitial atomic species diffuse more rapidly, CALLISTER (2013)^[32]. There are some factors that

influence diffusion and depends on both the diffusing host component and the temperature, this diffusion coefficient being a function of temperature. In semiconductors, heat treatments promote the diffusion of impurities that are transported into the host cell and it may also occur that such transport carries these impurities further into the host cell, generating a more suitable concentration distribution.

The diffracto grams show formation of the TTB structure with monophasic and crystalline characteristics even with doping's made with the nickel metal forming the system $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15,\delta}$ where x varies its doping, $x = 0,25; 0,50$ and $0,75$. The calcination at 1250°C , the grains present expansion evidenced in the diffracto graphic peaks exposed in Figure 2, where these peaks are pointed and narrow, what causes the system to present crystallinity and to be monophasic. This process favors the absence or even decrease (elimination) of defects found in the crystalline lattice due solely to structural homogeneity. By means of this phase

expansion, we have an increase in crystallinity, which can be evidenced by decreasing the width of the diffraction peaks, increasing in intensity and in numbers. The diffractogram (Figure 2) shows the formation of the phase with structure of tetragonal symmetry (TB). With the thermal treatment of the precursor powder performed at 1250°C / 1h the values of the interplanar distances, relative intensity and 2θ of the experimental diffractogram coincide with the crystallographic JCPDS_34-0108 (2000)^[27], this sheet refers to the phase of $\text{KSr}_2\text{Nb}_5\text{O}_{15}$. No other secondary phase has been identified, showing that the compound obtained is monophasic. For solid solutions with $x \leq 0,75$ diffractograms showed the formation of a single phase associated with $\text{KSr}_2\text{Nb}_5\text{O}_{15}$.

The valence of the Ni^{2+} in solid solutions of $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$, since the oxidation state 3+ of the nickel (Ni) cation has been rarely detected. The substitution of cations of large radius (r) such as, for example, Sr^{2+} ($r_{\text{Sr}^{2+}} = 1,18 \text{ \AA}$) by cations of small radius (r), such as Ni^{2+} ($r_{\text{Ni}^{2+}} = 0,69 \text{ \AA}$) not favorable. In addition, the cations of Ni^{2+} show strong preference for octahedral coordination, the same coordination of niobium (Nb). In this sense, the ionic radius of Nb cations in a high oxidation state ($r_{\text{Nb}^{5+}} = 0,64 \text{ \AA}$), similarity with the ionic radius of the Ni^{2+} . However, the valence difference is equal to three units, which is not favorable. However, the best similarity of the ionic Ni^{2+} occurs for the Nb^{5+} partially reduced to Nb^{4+} ($r_{\text{Nb}^{4+}} = 0,68 \text{ \AA}$). Here it is important to note that the Nb4 + represents a partial reduction of the niobium cation, whereas a completely reduced state is given by the niobium with valence 3+, Nb^{3+} , ($r_{\text{Nb}^{3+}} = 0,72 \text{ \AA}$); LANFREDI et al. (2005)^[26]. The considerable increase in the network parameters may be associated with the partial substitution of ions Nb^{5+} by the ions Ni^{2+} in the crystalline lattice, where there is a decrease in the covalent character of the bond Nb-O with the increase in the ionic character of the bond Ni-O; LANFREDI et al. (2015)^[33]. In fact, the results of the FT-IR (Fig. 1) showed a correlation between the wave number of the bands and the displacement magnitude of the decentralized Nb location as a function of the increased doping of the powders $\text{KSr}_2\text{Nb}_5\text{O}_{15}$; LANFREDI, FOLGUERAS-DOMÍNGUES, RODRIGUES (1995)^[25]. The partial replacement of niobium cations by nickel cations in the host structure can be accompanied by the formation of oxygen vacancies from the charge compensation mechanism due to the partial reduction of niobium; LANFREDI et al. (2015)^[33]. Thus, the formation of oxygen vacancies can be accompanied by disproportionation of cations Nb^{5+} for Nb^{3+} , in which it presents a larger ionic value due to its punctual loading, where $r_{\text{Nb}^{3+}} > r_{\text{Ni}^{2+}} > r_{\text{Nb}^{5+}}$, thus justifying the decrease in the volume value of the unit cell. Furthermore, nickel cations specifically occupy the position of the Nb(1). This may be due to the

ion ray value and the preferential octahedron occupation. This occupation results in a degree of distortion of the octahedron of the $[\text{Nb}(2)\text{O}_6]$, where this distortion is necessary for an accommodation of the nickel cations to occur in the formation of the structure.

In the host structure $\text{KSr}_2\text{Nb}_5\text{O}_{15}$, the niobium has preference in occupying the octahedral sites, NbO_6 . Cation substitution Nb^{5+} by cations Ni^{2+} can cause distortions in the octahedra as well as the creation of a sub level of energy resulting from the formation of gaps due to the difference of electrons between the cation Nb^{5+} and doping cation, resulting in a decrease in the band gap. A higher degree of distortion and structural defects can be expected as a result of increased nickel doping. However, doping with 0.75 mol of Nickel can cause such a disorder, in such a way that the electronic mobility of the electrons is hampered, requiring a greater energy for electronic transitions of the conduction band to the valence band.

In terms of the first-order apparent rate, it should be noted that all niobate-based materials presented less photo activity when compared to TiO_2 . The conversion of Basic Blue 41 is probably attributed to a series of chemical reactions occurring on the outer surface of these doped Nb-based materials Ni^{2+} . Thus, a deeper analysis of the photo catalytic activity can be performed to obtain the overall reaction rate considering the surface concentration of Basic Blue 41, since this primary fraction of Basic Blue 41 molecules will undergo photo degradation. The photo catalytic degradation of Basic Blue 41 can be considered as a unimolecular catalytic surface reaction, where the adsorption of Basic Blue 41 followed by photo degradation under irradiation UV; ATAR, OLGUN, ÇOLAK (2008)^[1]. The reaction rate for the degradation of Basic Blue 41 was faster for photo catalysts with $\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$ where $x = 0,50$ ($\text{KSr}_2\text{Ni}_{0,50}$). These results suggest that changes in the crystalline structure of the niobate-based material, especially when increased binding occurs Nb-O(6) can notably influence photo catalytic activity. Probably, it is the most active oxygen in the sense of probability of the transfer of charge to the molecules of O_2 and therefore the longer the length of such a connection Nb-O the more this distortion occurs, and thus the greater the efficiency of electron transfer and the greater the photo catalytic activity. Otherwise, it can serve as a more active center, a trap; JUAN, JORGE, JEAN-MARIE (2001)^[34] and JUAN, JORGE, JEAN-MARIE (1998)^[35]^[34-35] on the nature of the dominant species of reactive oxygen.

V. CONCLUSÕES

The chemical synthesis based on the modified Polyol method proved to be adequate for the preparation of monophasic and crystalline post-

$\text{KSr}_2(\text{Ni}_x\text{Nb}_{5-x})\text{O}_{15-\delta}$, where $x = 0,25; 0,5$ and $0,75$ with stoichiometry can be controlled. In addition, the production of the post-ceramics with a shorter calcination time than those prepared by conventional mixing of oxides. Due to the high polarity of the polyols, the inorganic salts (precursors) are easily solubilized; The nucleation and growth steps occur at the boiling point of the polyalcohol (eg, ethylene glycol at 197°C , diethyleneglycol at 246°C and tetraethyleneglycol at 314°C) and the use of high temperatures (at the boiling point of the polyalcohol) produces materials with high crystallinity;

Absorption spectroscopy in the infrared region showed a displacement of the bands characteristic of the Nb-O for regions of low frequencies with increasing concentration of nickel cations. This displacement shows that the bonds between niobium and oxygen cations, for the nickel doped solid solutions, are shorter than $\text{KSr}_2\text{Nb}_5\text{O}_{15}$;

The lines of the crystal, JCPDS: 34-0108 (2000)[27], coincides with the peaks of the diffractogram, however it is noticed a singular difference of these peaks, relative intensity has lower intensity (figure 2 – c) $\text{KSNNi}_{0,75}$) in relation to the relative intensity of $\text{KSNNi}_{0,50}$ in Figure 2, where in this diffractogram the intensity of the peaks is much more expressive and defined, but narrower and sharper, which demonstrated in descending order of relative intensities $I_{R(\text{KSNNi}_{0,50})} > I_{R(\text{KSNNi}_{0,25})} > I_{R(\text{KSNNi}_{0,75})}$;

A decrease of the absorbance values as a function of time was observed for the concentration of the basic blue 41 dye in solution, resulting from the photo degradation of the photo catalytic tests performed for the photo catalyst $\text{KSNNi}_{0,50}$ which presented lower absorbance value.

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Comparative Analysis of Heavy Metal Removal using Activated Bamboo and Periwinkle Shell, A Case Study of Cr (III) ion

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Abstract- Heavy metal removal capacity of Activated Bamboo carbon (ABC) and Activated Periwinkle shell (APS) were investigated through adsorption process. ABC and APS were characterized using Fourier Transform Infrared (FT-IR), Energy dispersive X-ray (EDX) and Scanning Electron Microscopy (SEM). Parameters such as adsorbent dosage, pH of effluent solution, agitation speed and time of agitation were varied. Results obtained show that FT-IR spectrum of the activated carbon displays a number of absorption peaks, reflecting the complex bio-mass structure and a variety of functional groups such as -OH, -NH, C=O, C-H, C-N, CH₃ and CH₂ which explains its improved adsorption behaviour on the colloidal particles. Synthetic waste water was prepared and characterized for its pollution characteristics in terms of turbidity, pH, conductivity, total suspended solid, total dissolved solid, nitrate, Nitrite, Ammonium, sulphate, phosphate, dissolved oxygen, chemical oxygen demand (COD), dissolved oxygen, Biological Oxygen Demand (BOD), Cr, total aerobic count and total coli form count.

GJRE-C Classification: For Code: 090499



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Abstract- Heavy metal removal capacity of Activated Bamboo carbon (ABC) and Activated Periwinkle shell (APS) were investigated through adsorption process. ABC and APS were characterized using Fourier Transform Infrared (FT-IR), Energy dispersive X-ray (EDX) and Scanning Electron Microscopy (SEM). Parameters such as adsorbent dosage, pH of effluent solution, agitation speed and time of agitation were varied. Results obtained show that FT-IR spectrum of the activated carbon displays a number of absorption peaks, reflecting the complex bio-mass structure and a variety of functional groups such as -OH, -NH, C=O, C-H, C-N, CH₃ and CH₂ which explains its improved adsorption behaviour on the colloidal particles. Synthetic waste water was prepared and characterized for its pollution characteristics in terms of turbidity, pH, conductivity, total suspended solid, total dissolved solid, nitrate, Nitrite, Ammonium, sulphate, phosphate, dissolved oxygen, chemical oxygen demand (COD), dissolved oxygen, Biological Oxygen Demand (BOD), Cr, total aerobic count and total coli form count. The adsorption of Chromium, using Activated Bamboo carbon (ABC) and Activated Periwinkle shell (APS) has been modeled and studied statistically using a pure-quadratic statistical model and the model was adequate. Bamboo carbon showed higher removal efficiency than Periwinkle shell carbon in the range of 3-6%. The dosage of metal and agitation speed had the most significant effect on removal efficiency, with optimum dosage falling in the range of 12.9-16.2 mg/l, while range of optimum agitation speed could be from 138-200 rpm, which was the maximum used.

I. INTRODUCTION

Although industrialization is inevitable, various devastating ecological and human disasters which have continuously occurred over the last three decades or so implicate industries as major contributors to environmental degradation and pollution problems of various magnitudes. Rapid industrial developments in developed and developing countries have increased hazardous wastes generation several fold (Fridriksberg,1984).

Wastewater is any water that has been adversely affected in quality by anthropogenic influence. It comprises liquid waste discharged by domestic residences, commercial properties, industry, and/or

agriculture and can encompass a wide range of potential contaminants and concentrations. In the most common usage, it refers to the municipal wastewater that contains a broad spectrum of contaminants resulting from the mixing of wastewater from different sources. Jagadeesh, (2006) and Ademoroti, (1996).

Industrial and municipal wastewaters frequently contain varied concentrations of organics and heavy metal ions that can be harmful to aquatic life and human health. The conventional methods for treating such wastewaters are expensive. Consequently, the search for unconventional but effective and economical methods for the treatment and removal of organics and heavy metal ions has been on the increase in recent times. The use of natural wastes as adsorbents for organics and metal ions is being exploited because of their availability and low cost. Agricultural materials that have been investigated as adsorbents for metal ions in solutions include walnut waste (Randall et al, 1974), peanut skin (Randall et al, (1975), cotton (Roberts and Rowland, 1973), sugar cane waste (Kumar and Dara, (1992) rice straw (Marshall and Champagne, 1995), palm kernel shell (Ebhoaye and Oyesola (2004) and maize cob and cassava peels in the removal of organics (Ewansiha et al, (2005). Several researches have equally reported on the use of modified agricultural wastes in the sorption of metal ions from solutions (Okieimen et al, (1991); (Okuo and Ozioko, (2001); (Okieimen and Ebhoaye, (1986).

In general, there are various technological methods existing for the treatment of wastewater. Such methods include chemical precipitation, ion exchange, adsorption, membrane processes, supercritical fluid extraction, bioremediation and Oxidation with oxidizing agent. However, most of these technologies are either extremely expensive or not readily available. Efficient and environment friendly methods are thus needed to be developed for the treatment of wastewater. It has been observed that adsorption among others is a cost effective technique and easy to operate.

Heavy metals: It is often used as a group name for metals and semimetals (metalloids) that have been associated with contamination and potential toxicity or ecotoxicity. They have high utilities in industrial

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applications from papers to automobiles by their very characteristics properties. They are found in the deep bowels of the earth as ores. The metals are segregated from these ores, leaving behind the tailings that find their way into the environment as toxic pollutants. They get into the water bodies directly from point sources as sewage and non-point sources as runoff and through atmospheric deposition that are transported from long distances. Heavy metals affect every level of the food web, from producers in the trophic levels to the highest order carnivore by residing in the system and magnifying at every trophic status.

Bacteria count represent one aspect of water quality, it is a microbiological procedure which uses samples of water and from these samples determine the concentration of bacteria. This is an indicator of possible presence of pathogenic micro-organisms.

The number of pathogens in sewage contaminated water is a function of number of persons who excrete such organisms.

Biological characteristics: these have to do with the following:

- i. The principal group of micro-organism found in surface water and wastewater as well as those responsible for biological treatment
- ii. Pathogenic organisms found in wastewater
- iii. The organism used as indicator of pollution and their significance
- iv. The method used to enumerate the indicator organism
- v. The method used to enumerate the toxicity of treated waste water.

a) Adsorption

Adsorption is the accumulation of substances on the surface of a solid or liquid. The surface area of the adsorbent play an important role. The larger the surface area, the greater the extent of adsorption.

Adsorption is of two types, namely physical adsorption and chemical adsorption or chemisorption (Negi and Anad (2007)

The adsorbent that will be used in this research are Activated Carbons prepared from bamboo culms, and periwinkle shells. Activated carbon: This is also called activated charcoal. It is a form of carbon that has been processed to make it extremely porous and thus have a very large surface area available for adsorption or chemical reaction (CPL Caron link, (2008). The adsorptive property is due to its high degree of micro porosity in that just one gram of activated carbon has a surface area of approximately 500m² as determined typically by nitrogen gas adsorption. Sufficient activation for useful applications may come solely from the high surface area though further chemical treatment often enhances the adsorbing properties of the material. Activated carbon is usually derived from charcoal and are the most common adsorbents used for the removal

of organic compounds from air and aqueous waste streams (Tsai,Chang,and Lee 1997) . Any material with high carbon content and low in organics can be used as precursor in the production of activated carbon (Donnet et al, 1988). Literature survey indicates that there have been many attempts to obtain activated carbons or adsorbents from agricultural wastes such as coconut shells (Mortley et al, 1988, Lavne et al, 1989). Almond shell (Philip and Cigris, 1996, Gergov et al,1994) peanut hull, (Periasemy and Namasivayam,1995, Wafwoyo et al,1999), rice husk (Khahil, 1996) sugarcane bagasses (Girgis et al,1994) e.t.c.

II. STATISTICAL MODELS

a) Response Surface Methodology

In statistics, response surface methodology (RSM) explores the relationships between several explanatory variables and one or more response variables. The method was introduced by Box and Wilson (1951). The main idea of RSM is to use a sequence of designed experiments to obtain an optimal response. Box and Wilson suggest using a second-degree polynomial model to do this. They acknowledge that this model is only an approximation, but used it because such a model is easy to estimate and apply, even when little is known about the process (Asadi and Hamid, 2017)

Statistical approaches such as RSM can be employed to maximize the production of a special substance by optimization of operational factors. In contrast to conventional methods, the interaction among process variables can be determined by statistical techniques. Response surface methodology has an effective track-record of helping researchers improve products and services.

b) Analysis of variance

Analysis of variance (ANOVA) is a collection of statistical models used to analyze the differences among group means and their associated procedures (such as "variation" among and between groups), developed by statistician and evolutionary biologist Ronald Fisher. In the ANOVA setting, the observed variance in a particular variable is partitioned into components attributable to different sources of variation. In its simplest form, ANOVA provides a statistical test of whether or not the means of several groups are equal, and therefore generalizes the *t*-test to more than two groups. ANOVAs are useful for comparing (testing) three or more means (groups or variables) for statistical significance. It is conceptually similar to multiple two-sample *t*-tests, but is more conservative (results in less type I error) and is therefore suited to a wide range of practical problems. ANOVA is used in the analysis of comparative experiments, those in which only the difference in outcomes is of interest. The statistical significance of the experiment is determined by a ratio of

two variances. This ratio is independent of several possible alterations to the experimental observations. ANOVA is the synthesis of several ideas and it is used for multiple purposes. As a consequence, it is difficult to define concisely or precisely (Gelman, 2005).

c) *F-test*

An *F-test* is any statistical test in which the test statistic has an *F*-distribution under the null hypothesis. It is most often used when comparing statistical models that have been fitted to a data set, in order to identify the model that best fits the population from which the data were sampled. Exact "*F*-tests" mainly arise when the models have been fitted to the data using least squares. The name was coined by George W. Snedecor, in honour of Sir Ronald A. Fisher. Fisher initially developed the statistic as the variance ratio in the 1920s.

d) *T-test*

In statistics, the *t*-statistic is a ratio of the departure of an estimated parameter from its notional value and its standard error. It is used in hypothesis testing. Most frequently, *t* statistics are used in Student's *t*-tests, a form of statistical hypothesis testing, and in the computation of certain confidence intervals. The key property of the *t* statistic is that it is a pivotal quantity – while defined in terms of the sample mean, its sampling

Given: a function $f: A \rightarrow \mathbf{R}$ from some set A to the real numbers

Sought: an element x_0 in A such that $f(x_0) \leq f(x)$ for all x in A ("minimization") or such that $f(x_0) \geq f(x)$ for all x in A ("maximization").

Such a formulation is called an optimization problem or a mathematical programming problem (a term not directly related to computer programming, but still in use for example in linear programming). Many real-world and theoretical problems may be modeled in this general framework. Problems formulated using this technique in the fields of physics and computer vision may refer to the technique as energy minimization, speaking of the value of the function f as representing the energy of the system being modeled.

The function f is called, variously, an objective function, a loss function or cost function (minimization), a utility function or fitness function (maximization), or, in certain fields, an energy function or energy functional. A feasible solution that minimizes (or maximizes, if that is the goal) the objective function is called an *optimal solution* (Diewert et al., 2008).

In mathematics, conventional optimization problems are usually stated in terms of minimization. Generally, unless both the objective function and the feasible region are convex in a minimization problem, there may be several local minima. A *local minimum* x^* is defined as a point for which there exists some $\delta > 0$ such that for all x where the expression holds; that is to say, on some region around x^* all of the function values

distribution does not depend on the sample parameters, and thus it can be used regardless of what these may be.

e) *Optimization*

In mathematics, computer science and operations research, mathematical optimization, also spelled mathematical optimisation (alternatively named mathematical programming or simply optimization or optimisation), is the selection of a best element (with regard to some criterion) from some set of available alternatives.

In the simplest case, an optimization problem consists of maximizing or minimizing a real function by systematically choosing input values from within an allowed set and computing the value of the function. The generalization of optimization theory and techniques to other formulations comprises a large area of applied mathematics. More generally, optimization includes finding "best available" values of some objective function given a defined domain (or input), including a variety of different types of objective functions and different types of domains.

An optimization problem can be represented in the following way:

are greater than or equal to the value at that point. Local maxima are defined similarly. While a local minimum is at least as good as any nearby points, a global minimum is at least as good as every feasible point. In a convex problem, if there is a local minimum that is interior (not on the edge of the set of feasible points), it is also the global minimum, but a non-convex problem may have more than one local minimum not all of which need be global minima.

III. MATERIALS AND METHODS

a) *Adsorbent preparation and characterization*

Matured bamboo culms were harvested and cut into small strips with saw blade. Samples of 1kg each were weighed and heated to 500°C for three hours using the METM-525 Muffle furnace. The carbonized culms were then milled to fine powder, and sieved through a mesh size of 150 μ m. The carbon particles that passed through the screen were collected, characterized and designated as Bamboo culms carbon (BC).

The periwinkle shells were prepared to the desired biosorbents according to the method described by (Aisien et.al, 2014) with some modification. Thus, it was first washed with detergent solution and then with dilute HCl in order to remove soil debris. Then the clean

and dry parts were taken in a porcelain crucible and carbonized completely at 900 °C in a muffle furnace for 1 h. The cooled carbon was characterized by standard methods as described by (Ishak and Baker, 1995).

b) Preparation of Synthetic Wastewater

The mixed metal ions solution from cation (Cr²⁺) was prepared from analytical grade stock standard of concentration 1000 ppm. The solution of wastewater was introduced for each metal according to the method of continuous dilution from the sample solution. The pH of the wastewater was adjusted by using HNO₃ 1% (V/V) and/or NaOH. The final concentration of metal ions in wastewater was analyzed by atomic absorption spectrophotometer (AAS).

c) Analysis of Wastewater

All samples of the simulated waste water prepared above were analyzed as described in the standard methods for the Examination of water and

Linear Model:

$$y = a + bx_1 + cx_2 + dx_3 + ex_4 \tag{1}$$

Interaction Model:

$$y = a + bx_1 + cx_2 + dx_3 + ex_4 + fx_1x_2 + gx_1x_3 + hx_1x_4 + ix_2x_3 + jx_2x_4 + kx_3x_4 \tag{2}$$

Pure-quadratic model:

$$y = a + bx_1 + cx_2 + dx_3 + ex_4 + fx_1^2 + gx_2^2 + hx_3^2 + ix_4^2 \tag{3}$$

Quadratic model:

$$y = a + bx_1 + cx_2 + dx_3 + ex_4 + fx_1x_2 + gx_1x_3 + hx_1x_4 + ix_2x_3 + jx_2x_4 + kx_3x_4 + lx_1^2 + mx_2^2 + nx_3^2 + ox_4^2 \tag{4}$$

The model that best fits, based on the adjusted R-squared (closest positive value to 1), will be considered the statistical model that best describes the adsorption phenomenon and will be used for optimization to determine optimum conditions for the process.

The value of the R-squared will be used as a measure of model accuracy and the closer the values are to unity, the more accurate the model is. It will also be seen as a measure of variability in the data that the model explains. Its final acceptability will be judged in corroboration with the F-statistics p-value. The p-value of the F-statistics will be used as a measure of model adequacy and a value less than or equal to 0.05 will be acceptable based on 95% confidence interval. The significance of the main effects will be determined using Analysis of variance (ANOVA) which will be judged in a similar way to the F-statistics, using the p-value, while the statistical significance of each factor or their interactions in the model will be determined using the t-statistics value or its associated p-value. As a rule of thumb, T-statistics values with magnitude greater than or equal to 2 will indicate the significance of a factor or their interactions. The p-value of the T-statistics can be

wastewater APHA (1992) and the standard methods for water and effluents analysis, (Ademoroti, 1996).

d) Statistical modeling and optimization

The response surface (statistical) study and optimization will be done using the statistical toolbox of Design expert (version 6.0.6) software for statistical modeling, analysis of variance, surface response study and optimization. The study will be done for the experimental data using contact time (A), dosage (B), agitation speed (C) and pH (D) as factors, with the metal ion removal efficiency (y) as response. The same procedure will be applied to study and optimize the removal efficiency of Chromium (Cr), Lead (Pb) and Cadmium (Cd) contaminants using bamboo carbon (ABC) and periwinkle shell carbon (APS) respectively as adsorbents.

The experimental data (factors and response) will be fit to four response surface models for each adsorbent-adsorbate pair:

used alternatively in a similar way to the p-value of the F-statistics.

The interaction between the factors will be determined using the surface plots, especially its contour lines and corroborated with the numerical values from t-statistics while the optimization toolbox Design expert (version 6.0.6) will be used for the optimization of factors based on the model.

e) Characterization of Bamboo culms based activated carbon

Bamboo culms have been used to produce quality activated carbon, as other biomass, because of their inherent high densities and carbon content. In this study the carbon from the bamboo culms was prepared according to the method described by Ishak and Baker, 1999.

Some of the characteristics of the carbon obtained from bamboo culms are as presented in Table 1.

Table 4.1: Some characteristics of the activated bamboo carbon (ABC)

Characteristics	Values
pH of slurry at 28°C	6.3 ±0.1
Bulk density(g/ml)	0.67±0.01
Surface area(m ² /g)	660.37
Langmuir surface area (m ² /g)	3499
BJH method cum. adso. surface area	766
DH method cum. Adso. Surface area	827
DFT cum. Surface area	157
Pore volume cm ³ /g	38.21
Attrition(%)	37.1
Conductivity(Ω ⁻¹ m ⁻¹)	145.6±10.5
Loss on Ignition(%)	73.5±0.2

Negative surface charge: 1.92±0.01

The value of the pH of bamboo culms carbon (ABC) which is 6.3 is within the generally acceptable range or values (6-8) in many applications. Carbons with extreme pH values have been reported to be capable of increasing or decreasing pH of slurry to induce undesirable physical/ chemical changes; for instance, in cane sugar refining, carbon with low pH.

values could cause the inversion of sucrose to non-crystallizable sugars causing lower yields, while alkaline carbon could cause decolouration through alteration of molecular structure of sugar impurities. The value of pH of ABC reported here (6.3) is comparable with the values reported for rubber seed shell carbon (Okieimen et.al., 2005) and rice husk carbon (Okafor and Ujor, 1994).

Bulk density was determined by the tempering procedure described by (Ahmedina et.al, 1997). Bulk density is important when carbon is to be removed by filtration, because it determines that amount of carbon that can be contained in a filter of given solids capacity. Carbons with an adequate density also help to improve filtration rate by forming an even cake on the filter

surface (Ahmedina et.al, 1997). The values of bulk density of ABC reported in this study are well within the acceptable range for powdered activated carbon in many applications (B.D 0.67g/cm³) (Babayemi et.al, 2016).

The surface area of ABC was determined by the iodine adsorption method, which is a widely used routine procedure for the determination of surface area of powders. Large surface area is a requirement for good adsorbent. Surface area is a single most important characteristics of activated carbons designed for adsorption of compounds from liquid media. Report of previous studies (Okieimen et.al, 2005) that attempted to correlate surface area measurements by different methods suggests that the values of iodine number obtained for ABCC represent a fairly large surface area 79.7mg/g. The values of surface area of ABCC are comparable with those reported for rubber seed shell carbon and rice husk carbon (Okieimen et.al, 2005).

Characterization of Periwinkle shells carbon

Table 4.2: Some characteristics of periwinkle shells carbon (APS)

Characteristics	Values
pH of slurry at 28°C	6.5 ±0.2
Bulk density(g/ml)	0.23±0.05
Surface area(m ² /g)	318.84
Langmuir surface area (m ² /g)	5584.66
BJH method	766
DH method cum	827
DFT cum. Surface area	67.9
Pore volume(cm ³ /g)	0.16
Attrition (%)	25.5±0.1
Conductivity(Ω ⁻¹ m ⁻¹)	111.5±12.2
Loss on Ignition (%)	51.0±0.5

Negative surface charge 1.79±0.02

The surface area of periwinkle shell carbon (APS) was determined by the iodine adsorption method: This is a widely used routine procedure for the

determination of surface area of powders. Large surface area is a requirement for good adsorbent. Surface area is a single most important characteristics of activated

carbons designed for adsorption of compounds from liquid media. Report of previous studies (Okieimen et.al, 1991) that attempted to correlate surface area measurements by different methods suggests that the values of iodine number obtained for APS represent a fairly large surface area. The values of surface area of APS are comparable with those reported for rubber seed shell carbon (Okieimen et.al, 2005) and rice husk carbon (Okieimen et.al, 2005; Ejikeme et.al, 2014)

f) Surface Charge

The adsorption capacity of carbons is determined by their porous structure and surface area but is also strongly influenced by the presence of functional groups at the surface. Activated carbons are known to contain a variety of hetero atoms such as oxygen, hydrogen, chlorine and Sulphur. Heteroatom's are either derived from the starting material, and become a part of the chemical structure as a result of imperfect carbonization, or chemically bonded to the carbon during activation or during subsequent additional treatments, such as oxidation. These heteroatom's are bound to the edges of the carbon layers and form surface groups that greatly affect the adsorption behavior of the activated carbon, with carbon-oxygen surface structures being by far the most important in influencing surface characteristics (Bansal et.al, 1988; Boehm, 1994).The most common are carboxyl, carbonyl, phenolic hydroxyl, anhydride, ether-type, lactones and lactal. The presence surface oxygen complexes imparts a polar character to the activated carbon surface, which should affect preferential adsorption of polar organic solutes.

Loss on ignition of carbon is the residue that remains when the carbonaceous portion is burned off. The ash consists mainly of minerals such as silica, aluminum, iron, magnesium and calcium. Loss of ignition in activated carbon is not desirable and is considered an impurity. Ash leached into sugar liquor during the process of decolorisation is known to cause uneven distribution of heat in the boiler during sugar crystallation. Loss of ignition may also interfere with carbon adsorption through competitive adsorption and catalysis of adverse reactions. For instance, the loss of ignition may affect the pH of the carbon since the pH of most commercial carbons is provided by their inorganic components. Usually, materials with the lowest loss of ignition produce the most active products.

The value obtained for the carbon is 51.0%; this result is an indication of the level of impurity present in it. (Zadok, 1985; Meites, 1963).

Bulk density is important when activated carbon is to be removed by filtration, because it determines that amount of carbon that can be contained in a filter of given solids capacity. Carbons with an adequate density also help to improve filtration rate by forming an even cake on the filter surface (Ahmedna et.al, 2000). The values of bulk density of APS reported in this study are well within the acceptable range for powdered activated carbon in many applications (B.D 0.23g/ml), Table 4.2. The values of the pH of APS are within the generally acceptable range or values (6-8) in many applications, Table 4.2

Table 4.3

Parameters	Units	Wastewater	FEPA limits	WHO limits (WHO,1973)
Turbidity	NTU	027.33±2.00	5.00	5
pH		8.10±0.01	6.00-9.00	6.5 to 8.5
Colour	Hazen	Blueish	-	15
Odour		Slightly offensive		Unobjectable
Temperature	0C	37.00±0.10	<40.00	10-30
Conductivity	µs/cm	935.00±2.00	2500.00	
TSS	mg/l	033.00±1.00	30.00	10
TDS	mg/l	445.50±1.45	2,000.00	1000
TS	mg/l	478.20±0.01	2030.00	500
NO ₃ -N	mg/l	13.00±0.01	20.00	-
NO ₂ -N	mg/l	0.00±0.00	0.50	-
NH ₄ -N	mg/l	0.00±0.00	0.50	-
Sulphate	mg/l	109.00±0.02	500.00	250
Phosphate	mg/l	09.11±0.11	5.00	-
COD	mg/l	115.00±1.00	80.00	
DO	mg/l	0.45±0.01	5.00	4
BOD	mg/l	00.00±0.00	30.00	3
Pb	mg/l	31.68±0.02	<1.00	0.01
Ni	mg/l	50.00±0.02	<1.00	0.02
Zn	mg/l	45.05±0.01	<1.00	3
Cu	mg/l	32.5.±0.05	<1.00	2
Cr	mg/l	45.00±0.01	<1.00	0.05

Total Aerobic Cnt	Cfus/ml	Nil	400MNP/100ml	5000
Total Coliform Cnt	Cfus/ml	NIL	400MNP/100ml	

Result of characterization of Waste Water (synthesized)

Table 4.4: Characterization of the wastewater after treatment with Bamboo carbon (ABC) and periwinkle shells carbon (APS)

Parameters	Units	Wastewater	Treatment with ABS	Treatment with APS
Turbidity	NTU	27.32±2.00	08.00±0.02	16.11±0.01
P ^H		8.10±0.00	7.12±0.00	7.80±0.00
Colour	Hazen	Blueish	Colourless	Colourless
Odour		Slightly offensive	Odourless	Odourless
Temperature	0C	27.00±0.10	27.00±0.00	27.00±0.00
Conductivity	µs/cm	935.00±2.00	385.00±2.00	403.05±0.01
TSS	mg/l	33.00±1.00	08.00±0.01	18.00±0.01
TDS	mg/l	445.50±1.00	189.50±0.02	225.00±0.01
TS	mg/l	478.50±2.00	197.50±0.03	243.00±0.01
NO ₃ -N	mg/l	13.00±0.10	BDL	BDL
NO ₂ -N	mg/l	NIL	NIL	NIL
NH ₄ -N	mg/l	NIL	NIL	NIL
Sulphate	mg/l	109.00±2.00	46.09±0.01	53.30±0.01
Phosphate	mg/l	09.01±0.10	1.44±0.01	1.45±0.00
COD	mg/l	115.00±2.00	10.50±0.00	32.00±0.02
DO	mg/l	0.45±0.01	1.92±0.41	0.85±0.08
BOD	mg/l	00.00±0.00	0.00±0.00	0.00±0.00
Pb (II)	mg/l	31.68±0.02	1.26±0.02	3.17±0.01
Ni (II)	mg/l	50.00±0.02	4.5±0.02	7.5±0.01
Zn (II)	mg/l	45.00±0.01	0.09±0.01	6.75±0.01
Cu (II)	mg/l	32.5±0.02	1.95±0.01	3.58±0.02
Cr (III)	mg/l	45.00±0.01	6.30±0.02	9.00±0.01
Cd (II)	mg/l	10.00±0.01	0.20±0.01	0.50±0.00
Total Aerobic Cnt	Cfus/ml	NIL	NIL	NIL
Total Coliform Cnt	Cfus/ml	NIL	NIL	NIL

g) Adsorption Isotherms Studies

Two important physiochemical aspects for the evaluation of the adsorption process as a unit operation are equilibria of the adsorption and the kinetics. Equilibria studies give the capacity of the adsorbent.(Olafadehan and Aribike,2000)

The equilibrium relationships between adsorbent and adsorbate are described by adsorption isotherms, usually the ratio between the quantity

adsorbed and that remaining in solution at a fixed temperature at equilibrium.

Adsorption isotherms are characterized by certain constants and described the mathematical relationship between the quantity of adsorbate and concentration of adsorbate remaining in the solution at equilibrium. In this work, Langmuir, Freundlich and Temkin isotherm models have been used to analyze adsorption data at different concentrations.

Langmuir adsorption isotherm:
$$\frac{a_L C_e}{1 + b_L C_e}$$

i

Freundlich adsorption isotherm:
$$q_e = k_f C_e^{1/n}$$

ii

Linearising equation I and ii gives
$$\frac{C_e}{q_e} = \left(\frac{1}{a_L} \right) + \frac{b_L}{a_L} C_e$$

iii

$$\ln q_e = \ln k_f + \frac{1}{n} \ln C_e$$

iv

Table 4. 6: Isotherm data for Chromium

Co = 45mg/l						Cr				
B	E(ABC)	Ce(ABC)	Qe(ABC)	1/Ce(ABC)	1/Qe (ABC)	E(APS)	Ce(APS)	Qe(APS)	1/Ce(APS)	1/Qe (APS)
1	40.5	26.78	18.23	0.0373	0.0549	23.4	34.47	10.53	0.0290	0.0950
5	46.8	23.94	4.212	0.0418	0.2375	34.8	29.34	3.132	0.0340	0.3193
10	61.5	17.33	2.768	0.05772	0.3613	42.5	25.88	1.9125	0.0386	0.5229
15	84.1	7.155	2.523	0.1398	0.3964	75.4	11.07	2.262	0.0903	0.4421
20	62.4	16.92	1.404	0.0591	0.7123	50.6	22.23	1.1385	0.0449	0.8783
25	48.5	23.18	0.873	0.0432	1.1455	40.5	26.78	0.729	0.0373	1.3717

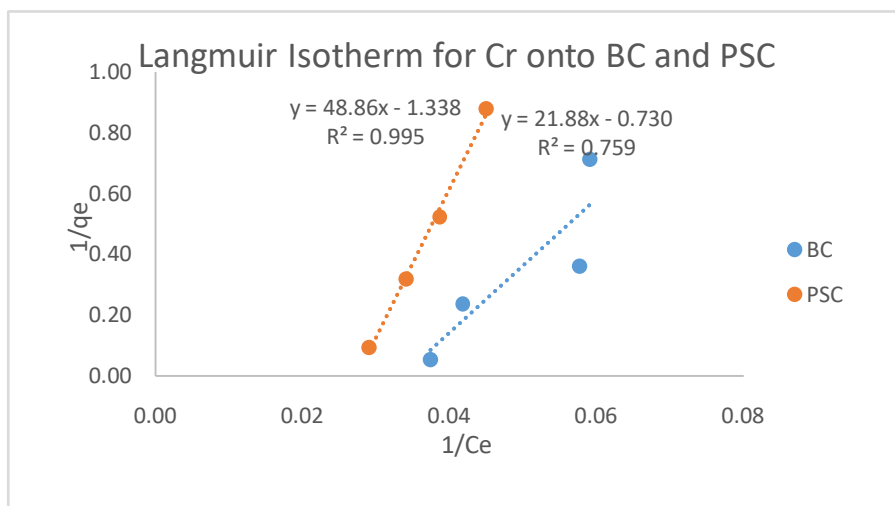


Fig. 4.4: Langmuir Isotherm for Cr adsorption onto ABC and APS

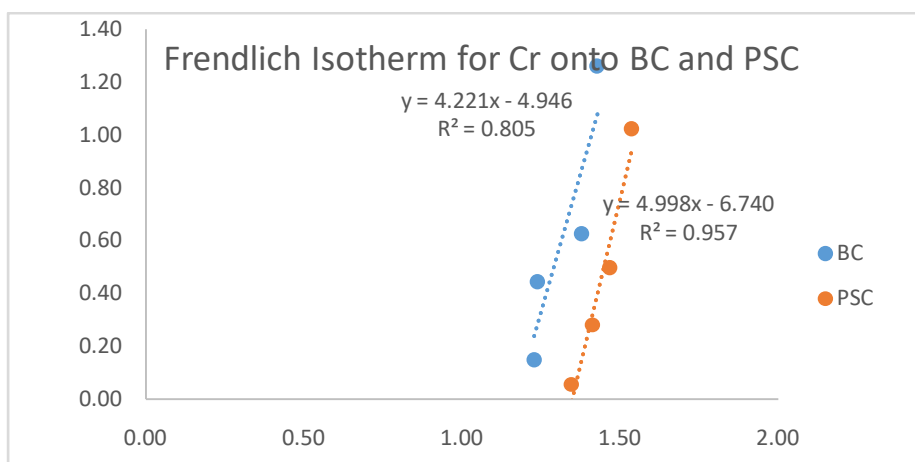


Fig. 4.5: Freundlich Isotherm for Cr adsorption onto ABC and APS

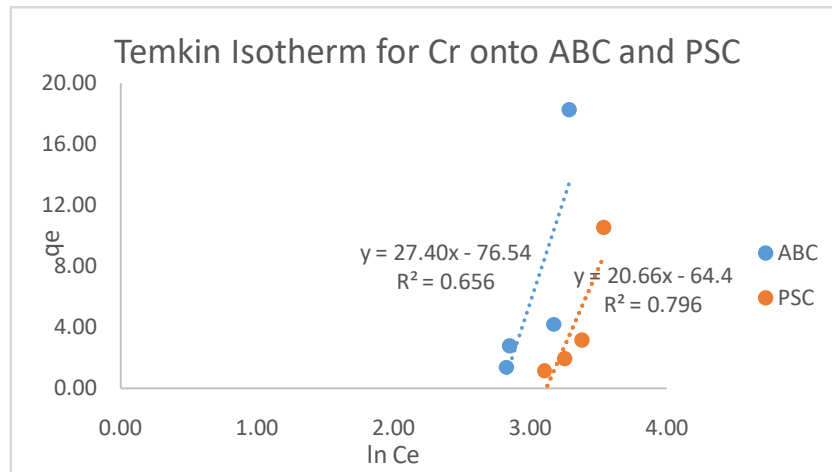


Fig. 4.6: Temkin Isotherm for Cr adsorption onto ABC and APS

h) Sorption Kinetics Studies for Cr³⁺ for Bamboo Carbon and Periwinkle Shell Carbon

Except for sorption capacity, kinetic performance of a given bio sorbent is also of great significance for the pilot application. From the kinetic analysis, the solute uptake rate, which determines the

residence time required for completion of sorption reaction, may be established. Also, one can know the scale of sorption apparatus based on the kinetic information. Generally speaking, sorption kinetics is the base to determine the performance of fixed-bed or any other flow-through systems.

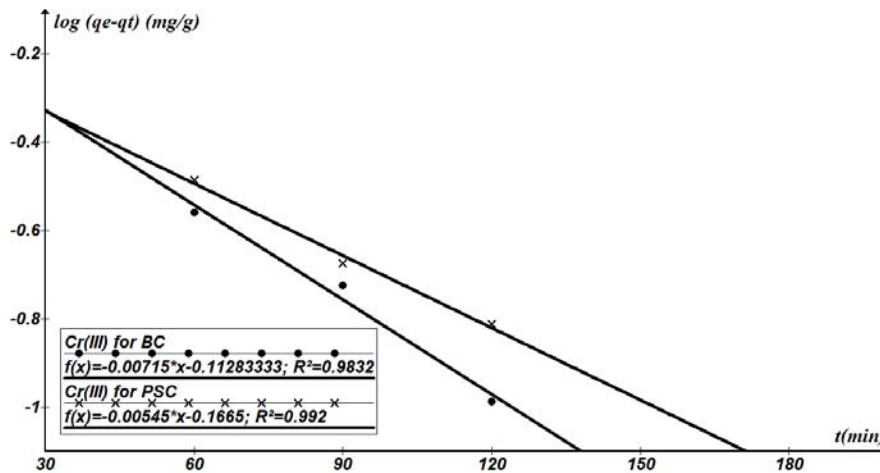


Fig. 4.11: Pseudo first order for ABC and APS for the sorption of Cr(III) ions

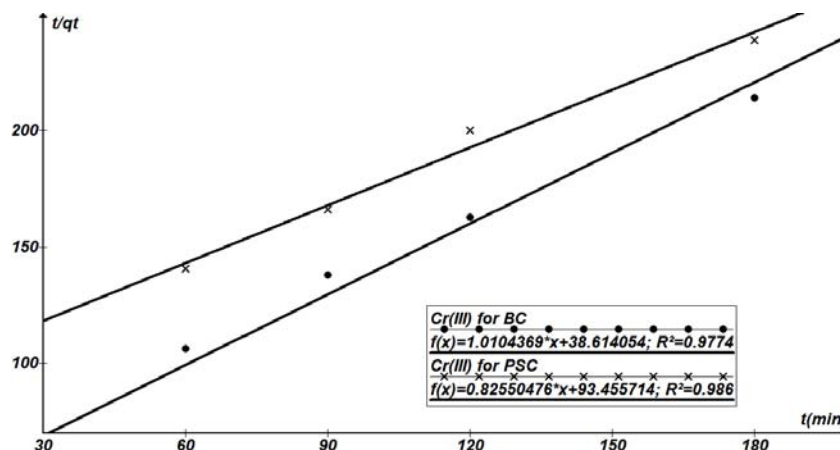


Fig. 4.14: Pseudo Second order for ABC and APS for the sorption of Cr(III) ions

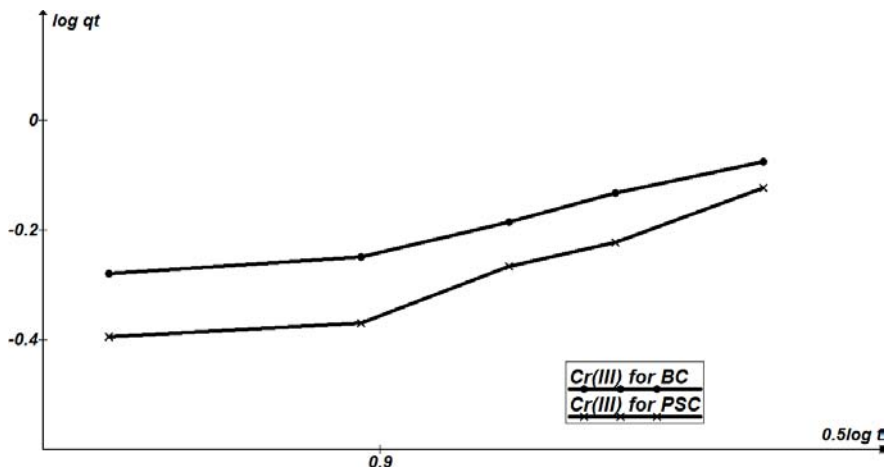


Fig. 4.17: Intraparticle diffusion model for ABC and APS for the sorption of Cr(III) ions

The results obtained from the different studies of the sorption of Cr^{3+} by ABC and APS in Table 4.9 show the affinities of the biosorbents for the metal ions. The kinetic properties of the sorbate uptake are required for selecting optimum operating conditions for full scale batch process (Gupta et al, 2010). The kinetic parameter, which is helpful for the prediction of sorption rate, gives important information for designing and modelling sorption processes.

Kinetic models can be helpful to comprehend the mechanism of metal sorption and estimate performance of the sorbents for metal removal. In order to further determine the mechanism of Cr^{3+} sorbed onto the biosorbents (ABC and APS), several kinetic models were exploited to discern the controlling mechanism. They included the pseudo first order, pseudo second order, and the intra particle diffusion models. The conformity between the experimental data and the model predicted values were expressed by coefficients of determination (R^2).

Different kinetic parameters calculated by linear regression for Cr^{3+} initial concentrations are summarized in Table 4.9. Plots of pseudo first order, pseudo second order, and intraparticle models are shown in Figures 4.11-4.18. As shown in Table 4.9, the coefficients of determination (R^2) obtained from the plots of pseudo

first and second orders kinetics were higher ($R^2 > 0.9$) for both ABC and APS, than those of the intraparticle model.

It can also be seen in Table 4.9 that, the rate constant (k_2) of ABC is higher for both Cr^{3+} . A similar observation has also been reported by earlier researchers (Allen et al., 2005, Liu et al., 2012).

The results of the sorption of Cd^{2+} , Pb^{2+} and Cr^{3+} on to the biosorbents have been represented in the form of the pseudo first order equation in Figures 4.10 to 4.12. From these plots, a linear relationship between $\ln(q_e - qt)$ and t was established with high correlation coefficients ($R^2 = 0.9$) for Cd^{2+} and Cr^{3+} with both ABC and APS and ($R^2 = 0.98$ & 0.96) for Pb^{2+} for both ABC and APS respectively. This show that the model can be applied and is appropriate to described the entire process. These plots showed different distinct linear regions within individual sets of data also for the pseudo second order model.

The applicability of the pseudo second order kinetic model to experimental data is presented in Table 4.9. The deviation between the calculated q_e values and the experimental q_e values were relatively low and a regression coefficient of 0.97-0.98 shows that the model can be applied for the sorption.

Table 4.9: Kinetic Parameters of the Kinetic Models for the Sorption of Cr^{3+}

Kinetic Model	Parameter	Cr^{3+}
Lagergren Pseudo first order	$q_e, \exp(\text{mg/g}), q_e, \text{calc}(\text{mg/g}), K_1 \times 10^{-3}(\text{hr}^{-1}), R^2$	0.841(0.754)0.771(0.682) 16.466(12.551)0.9832(0.9920)
Pseudo Second Order	$q_e, \text{calc}(\text{mg/g}), K_2 \times 10^{-2}(\text{hr}^{-1}), R^2$	0.990(1.211)1.321(0.365) 0.9774(0.9860)
Intraparticle Diffusion Model	$K_{id}(\text{mg/ghr}^{-1/2}), \text{Intercept}, R^2$	0.1107(0.1988)-0.9556(-0.7015)0.9114(0.9352)

ABC = Activated Bamboo carbon, APS = Activated Periwinkle shell carbon (in parenthesis)

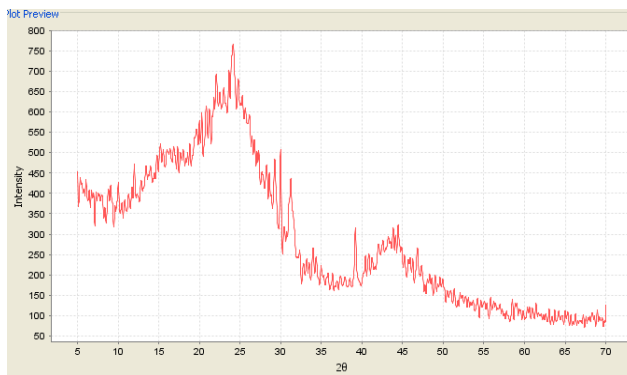


Fig. 4.19: X-ray diffraction (XRD) of ABC

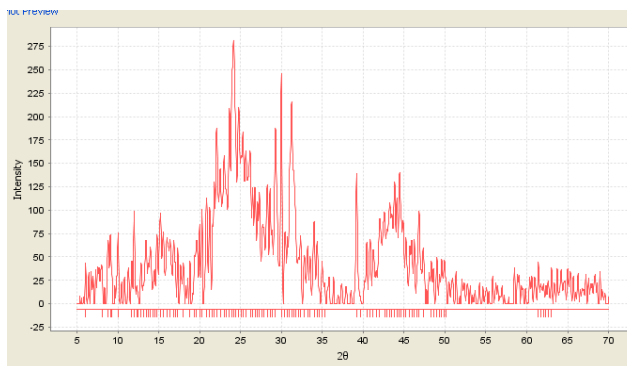


Fig. 4.20: X-ray diffraction (XRD) of APS

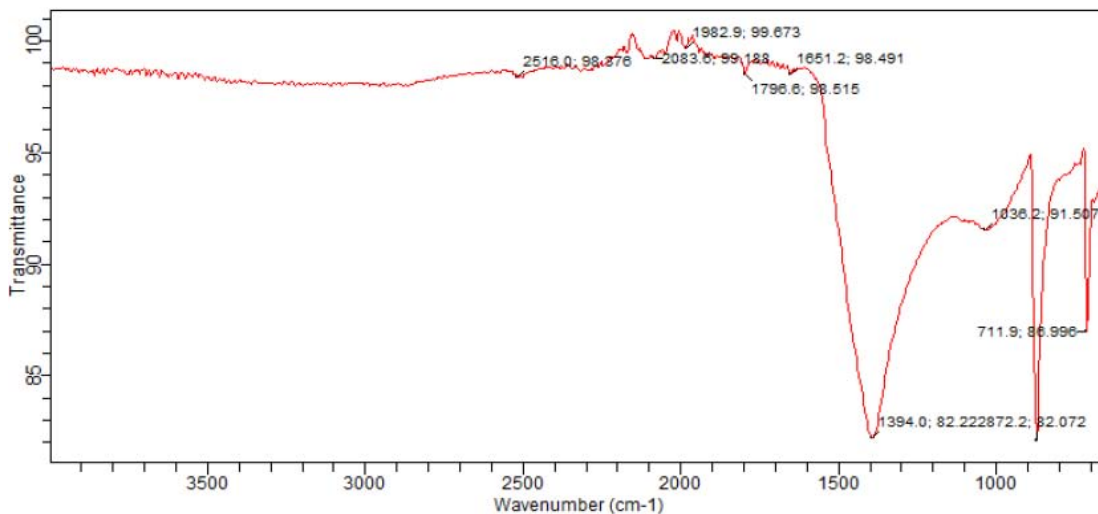


Fig. 4.21: FT-IR spectrum of APS

Fourier Transform Infrared Spectroscopy was carried out in order to identify the functional groups present in activated carbon. Functional groups of adsorbents not only affect the adsorption behavior but also dominate the adsorption mechanism. The spectra of adsorbent were measured in the range of 4000cm⁻¹ to 500cm⁻¹ wave number. The FT-IR spectrum reveals the complex nature of the adsorbent as evidence by the presence of a large number of peaks. The FT-IR spectrum of ABC as shown in the figure 4.21, shows

absorption bands at 2516 /cm, a carboxylic O-H and S-H thiols, a C-C triple bond at 2084 /cm, a C=O (acyl and halides) at 1797/cm, an alkene C=C, amines and amides at 1651/cm, a C-O-C at 1036 /cm, a C-C single bond (alkane functional group) at 872/cm and -S-OR (esters) at 712/cm. All these show that APS, with so many functional groups on it will be able to attract most of the cations desired to be removed from the waste effluent.

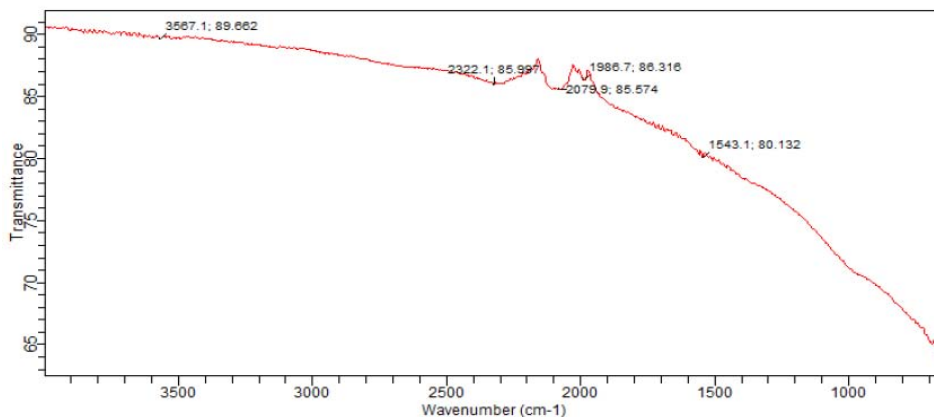


Fig. 4.22: FT-IR Spectrum of ABC

The FTIR spectrum of APS in the figure 4.22 gives absorption bands at 3567/cm, an –OH broad and single –Si-H stretching at 2333/cm, a C-C triple bond showing the alkyne group at 2080/cm and –Ntro ,N-H,C-

C double bond at 1543/cm showing the aromatic functional group

Table 4.10: Data for statistical study and optimization of Chromium ion removal at 30°C

Std	Run	Block	Factor 1 A:Contact time min	Factor 2 B:Dosage	Factor 3 C:Speed	Factor 4 D:pH	Response 1 Removal effici %	Response 2 Removal effici %	
	5	1	Block 1	180.00	15.00	150.00	2.00	32.1	30.8
	7	2	Block 1	180.00	15.00	150.00	4.00	48.4	38.2
	4	3	Block 1	180.00	15.00	150.00	5.00	56.4	46.1
	11	4	Block 1	180.00	15.00	150.00	6.00	65.9	52.8
	13	5	Block 1	180.00	15.00	150.00	7.00	85.3	80
	1	6	Block 1	180.00	15.00	150.00	8.00	58.5	46.6
	16	7	Block 1	180.00	1.00	150.00	7.00	40.5	23.4
	10	8	Block 1	180.00	5.00	150.00	7.00	46.8	34.8
	3	9	Block 1	180.00	10.00	150.00	7.00	61.5	42.5
	8	10	Block 1	180.00	15.00	150.00	7.00	84.1	75.4
	14	11	Block 1	180.00	20.00	150.00	7.00	62.4	50.6
	12	12	Block 1	180.00	25.00	150.00	7.00	48.5	40.2
	2	13	Block 1	180.00	15.00	0.00	7.00	40.5	23.4
	9	14	Block 1	180.00	15.00	50.00	7.00	56.8	44.8
	6	15	Block 1	180.00	15.00	100.00	7.00	71.5	62.5
	15	16	Block 1	180.00	15.00	150.00	7.00	84.1	75.4
	17	17	Block 1	180.00	15.00	200.00	7.00	72.4	55.6
	18	18	Block 1	30.00	15.00	150.00	7.00	52.6	40.3
	19	19	Block 1	60.00	15.00	150.00	7.00	56.4	42.7
	20	20	Block 1	90.00	15.00	150.00	7.00	65.2	54.2
	21	21	Block 1	120.00	15.00	150.00	7.00	73.8	60
	22	22	Block 1	150.00	15.00	150.00	7.00	80.1	68
	23	23	Block 1	180.00	15.00	150.00	7.00	85.4	71.6

Table 4.11: Coded factors in terms of alpha values Development of Models for Cr

Std	Run	Block	Factor 1 A:Contact time min	Factor 2 B:Dosage	Factor 3 C:Speed	Factor 4 D:pH	Response 1 Removal effici %	Response 2 Removal effici %
5	1	{ 1 }	1.000	0.167	0.333	-1.000	32.1	30.8
7	2	{ 1 }	1.000	0.167	0.333	-0.200	48.4	38.2
4	3	{ 1 }	1.000	0.167	0.333	0.200	56.4	46.1
11	4	{ 1 }	1.000	0.167	0.333	0.600	65.9	52.8
13	5	{ 1 }	1.000	0.167	0.333	1.000	85.3	80
1	6	{ 1 }	1.000	0.167	0.333	1.400	58.5	46.6
16	7	{ 1 }	1.000	-1.000	0.333	1.000	40.5	23.4
10	8	{ 1 }	1.000	-0.667	0.333	1.000	46.8	34.8
3	9	{ 1 }	1.000	-0.250	0.333	1.000	61.5	42.5
8	10	{ 1 }	1.000	0.167	0.333	1.000	84.1	75.4
14	11	{ 1 }	1.000	0.583	0.333	1.000	62.4	50.6
12	12	{ 1 }	1.000	1.000	0.333	1.000	48.5	40.2
2	13	{ 1 }	1.000	0.167	-1.667	1.000	40.5	23.4
9	14	{ 1 }	1.000	0.167	-1.000	1.000	56.8	44.8
6	15	{ 1 }	1.000	0.167	-0.333	1.000	71.5	62.5
15	16	{ 1 }	1.000	0.167	0.333	1.000	84.1	75.4
17	17	{ 1 }	1.000	0.167	1.000	1.000	72.4	55.6
18	18	{ 1 }	-1.000	0.167	0.333	1.000	52.6	40.3
19	19	{ 1 }	-0.600	0.167	0.333	1.000	56.4	42.7
20	20	{ 1 }	-0.200	0.167	0.333	1.000	65.2	54.2
21	21	{ 1 }	0.200	0.167	0.333	1.000	73.8	60
22	22	{ 1 }	0.600	0.167	0.333	1.000	80.1	68
23	23	{ 1 }	1.000	0.167	0.333	1.000	85.4	71.6

The functional relationship existing between the input and output variables were used to develop model equations. Experimental data were inputted into the design interface of the Design expert software and

hence generated polynomial model equations for the responses.

The following equations were generated using quadratic model of the software and were all in terms of coded factors as written.

$$\text{Removal efficiency (ABC)} = 53.96 + 12.20 * A + 7.54 * B + 6.96 * C + 23.20 * D - 8.41 * A^2 - 31.95 * B^2 - 7.56 * C^2 - 8.59 * D^2 \quad (R^2 = 0.561) \tag{1}$$

$$\text{Removal efficiency APS} = 43.32 + 12.74 * A + 11.58 * B + 4.30 * C + 18.44 * D - 7.57 * A^2 - 32.43 * B^2 - 11.76 * C^2 - 5.24 * D^2 \quad (R^2 = 0.9027) \tag{2}$$

IV. MODEL ADEQUACY CHECKING

After developing model equations, checking its adequacy is necessary for how properly fitted is the predicted data to the experimental data, to know the

errors or deviation known as residuals which can be studentized.

The statistical tools used were R^2 , R^2_{Adj} , $R^2_{Pred.}$, PRESS, Model P-value, Adequate precision, and Coefficient of variation (%) as shown in Table 4.12

Table 4.12: Regression Statistics for Checking Model Adequacy for Cr(III)

Statistics	Removal efficiency(%) ABC	Removal efficiency(%) APS
R^2	0.7917	0.7278
R^2_{Adj}	0.6726	0.5722
Model F-value	6.65	14.38
Model P-value (Prob.>F)	0.0011	0.0059
Adequate Precision	8.484	7.135
Coefficient of Var.	4.38	21.05

a) Model Validation using Graphical Approach

Validation of developed models is important to ensure that the models satisfactorily describe the behaviour of the system being modelled. Validation of models can be done using graphical or numerical approach. Graphical

method was used in this work to validate the developed models. As it is advantageous over other due to illustration of a broad range of relationship existing between model and data used. In Figures 4.23 and 4.24, the parity plot of predicted versus actual show that for the responses the points were randomly scattered

along 45 degrees line (straight line). Also, the normal probability plots of studentized residuals shown on Figure 4.23 and 3 show that residuals behave as expected and it implies that errors are evenly distributed

as the points fell on straight lines. These further imply that the models fit the data adequately (goodness of fit).

Removal efficiency of Cr by ABC, Response 1: Removal efficiency of Cr by ABC
ANOVA for Response Surface Quadratic Model
Analysis of variance table [Partial sum of squares]

Sum of Source	Mean Squares	F DF	Square Value	Prob > F		
Model	4247.37	8	530.92	6.65	0.0011	significant
A	674.06	1	674.06	8.44	0.0115	
B	166.80	1	166.80	2.09	0.1703	
C	126.95	1	126.95	1.59	0.2279	
D	1993.13	1	1993.13	24.97	0.0002	
A ²	99.17	1	99.17	1.24	0.2838	
B ²	1614.99	1	1614.99	20.23	0.0005	
C ²	183.64	1	183.64	2.30	0.1516	
D ²	158.81	1	158.81	1.99	0.1803	
Residual	1117.71	14	79.84			
Lack of Fit	1116.14	11	101.47	194.20	0.0051	
Pure Error	1.573	0.52				
Cor Total	5365.07	22				

Diagnostics Case Statistics table of Actual values vs predicted values for Cr using ABC
diagnostics Case Statistics

Standard Order	Actual Value	Predicted Value	Residual	Leverage	Student Residual	Cook's Distance	Outliert	Run Order
1	58.50	75.23	-16.73	0.480	-2.596	0.691	-3.474	6
2	40.50	40.12	0.38	0.846	0.107	0.007	0.103	13
3	61.50	69.95	-8.45	0.137	-1.018	0.018	-1.019	9
4	56.40	63.90	-7.50	0.316	-1.014	0.053	-1.015	3
5	32.10	27.81	4.29	0.929	1.800	4.703	1.978	1
6	71.50	69.56	1.94	0.255	0.252	0.002	0.243	15
7	48.40	54.62	-6.22	0.338	-0.856	0.042	-0.847	2
8	84.10	74.20	9.90	0.101	1.168	0.017	1.185	10
9	56.80	58.20	-1.40	0.314	-0.189	0.002	-0.182	14
10	46.80	54.60	-7.80	0.272	-1.024	0.044	-1.026	8
11	65.90	70.42	-4.52	0.189	-0.562	0.008	-0.548	4
12	48.50	49.42	-0.92	0.834	-0.253	0.036	-0.245	12
13	85.30	74.20	11.10	0.101	1.310	0.021	1.347	5
14	62.40	67.36	-4.96	0.176	-0.612	0.009	-0.597	11
15	84.10	74.20	9.90	0.101	1.168	0.017	1.185	16
16	40.50	34.34	6.16	0.735	1.339	0.553	1.382	7
17	72.40	72.13	0.27	0.756	0.061	0.001	0.059	17
18	52.60	49.80	2.80	0.809	0.718	0.243	0.705	18
19	56.40	60.06	-3.66	0.295	-0.488	0.011	-0.475	19
20	65.20	67.64	-2.44	0.350	-0.338	0.007	-0.327	20
21	73.80	72.52	1.28	0.371	0.181	0.002	0.175	21
22	80.10	74.71	5.39	0.197	0.674	0.012	0.660	22
23	85.40	74.20	11.20	0.101				
		1.321	0.022	1.361	23			

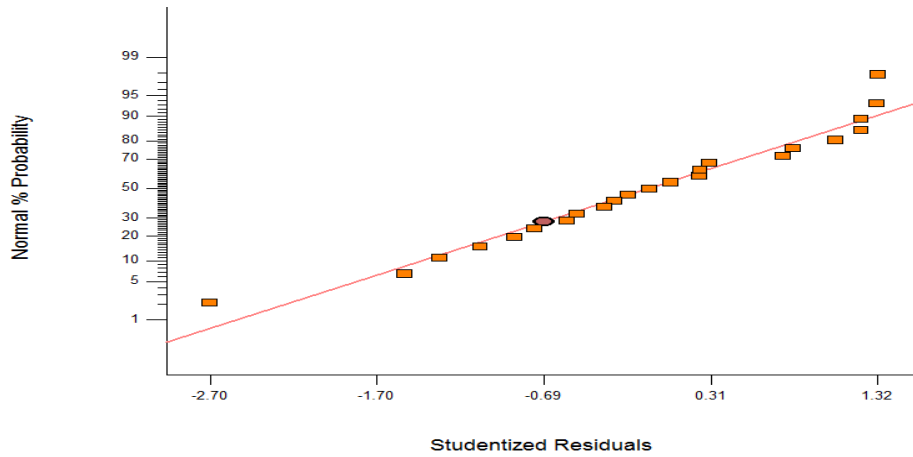


Fig.4.23: Normal probability plots of residuals for Removal Efficiency (ABC)

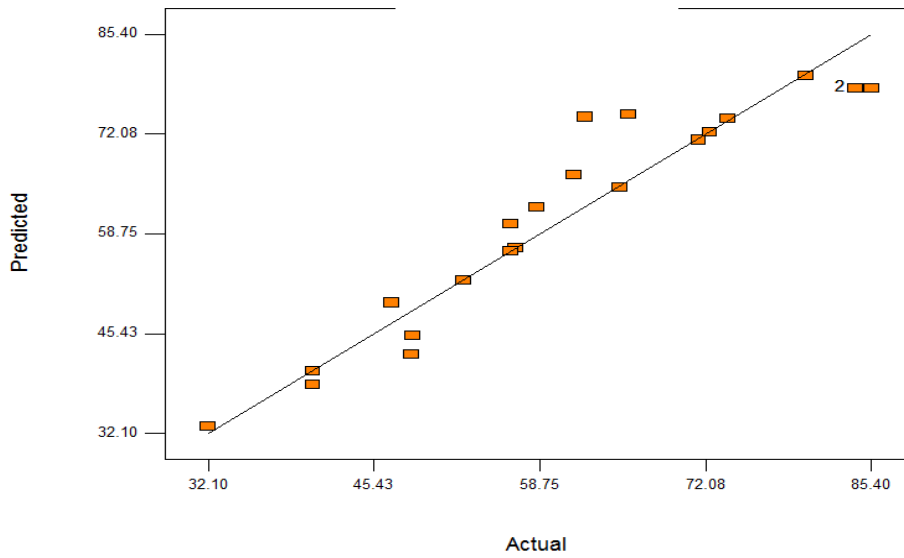


Fig.4.24: Predicted vs Actual values for Removal Efficiency (ABC)

DESIGN-EXPERT Plot

Removal efficiency
X = A: Contact time
Y = B: Dosage

Actual Factors
C: Speed = 125.00
D: pH = 4.50

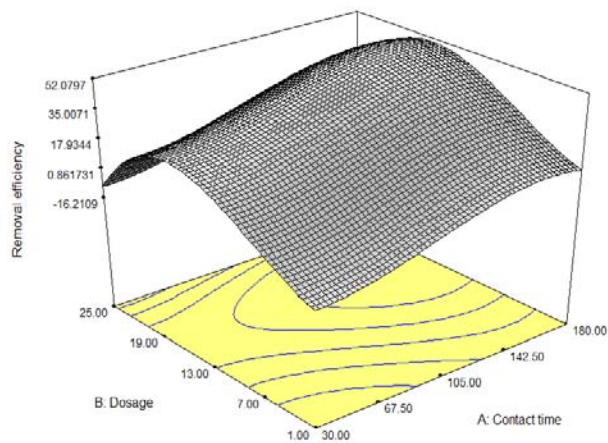


Fig.4.25: 3D Plot for the Effects of Contact Time (A) and Dosage (B) on the Removal Efficiency of ABC.

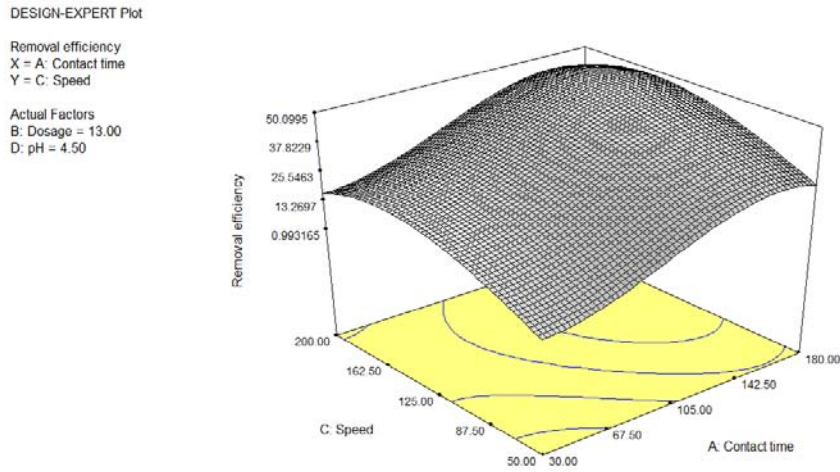


Figure 4.26: 3D Plot for the Effects of Contact Time (A) and Speed (C) on the Removal Efficiency of BC.

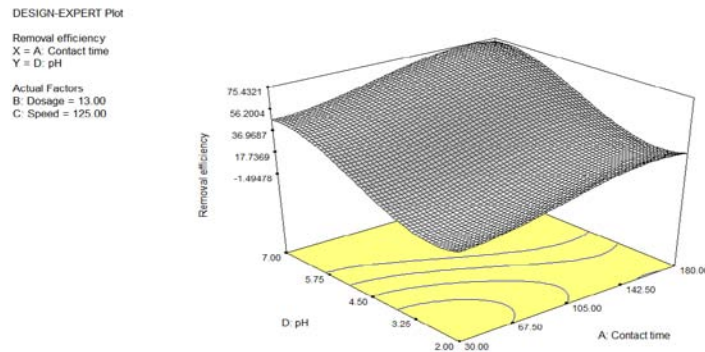


Figure 4.27: 3D Plot for the Effects of Contact Time (A) and pH (D) on the Removal Efficiency of ABC.

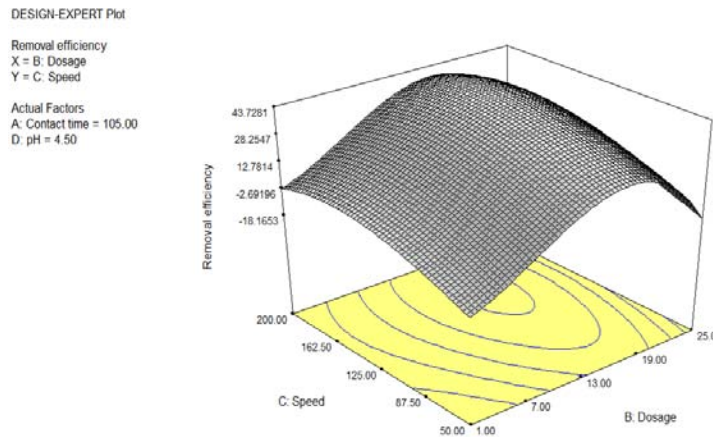


Fig.4.28: 3D Plot for the Effects of Dosage (B) and Speed (C) on the Removal Efficiency of BC.

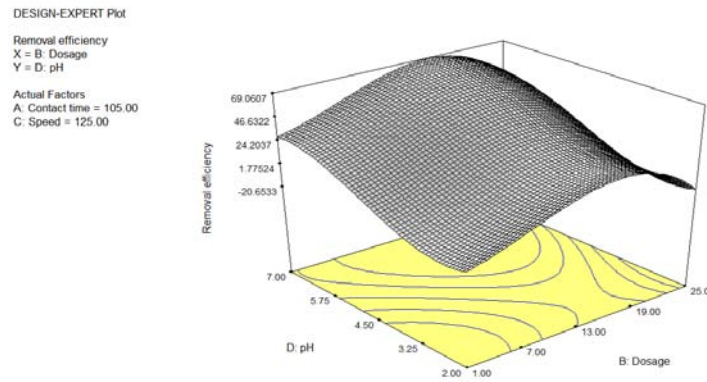


Fig.4.29: 3D Plot for the Effects of Dosage (B) and pH(D) on the Removal Efficiency of BC.

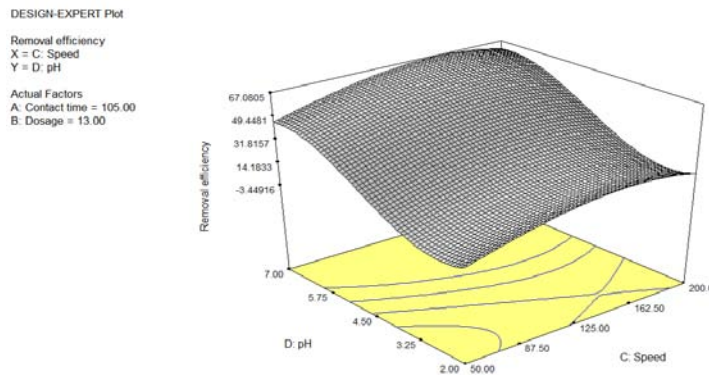


Fig.4.30: 3D Plot for the Effects of Speed (C) and pH(D) on the Removal Efficiency of BC.

Solutions Number	A	B	C	D	Removal efficiency	Desirability
1	<u>160.06</u>	<u>16.62</u>	<u>155.38</u>	<u>6.74</u>	<u>81.961</u>	<u>0.935</u> Selected

Table 4.16: ANOVA for Response Surface Quadratic Model for Removal efficiency of Cr by APS
Analysis of variance table [Partial sum of squares]

Sum of Source	Mean Squares	FDF	Square	Value	Prob > F
Model	4219.21	8	527.40	4.68	0.0059 significant
A	735.06	1	735.06	6.52	0.0230
B	393.45	1	393.45	3.49	0.0828
C	48.45	1	48.45	0.43	0.5227
D	1259.10	1	1259.10	11.17	0.0048
A ²	80.27	1	80.27	0.71	0.4130
B ²	1663.79	1	1663.79	14.76	0.0018
C ²	444.51	1	444.51	3.94	0.0670
D ²	59.00	1	59.00	0.52	0.4813
Residual	1578.38	14	112.74		
Lack of Fit	1564.28	12	130.36	18.48	0.0524 not significant
Pure Error	14.11	2	7.05		
Cor Total	5797.59	22			

Table 4.17: Diagnostics Case Statistics table of Actual values vs predicted values for Cr using APS.
Diagnostics Case Statistics

Standard Run Order	Actual Value	Predicted Value	Residual	Leverage	Student Residual	Cook's Distance	t	Outlier Order
1	46.60	65.19	-18.59	0.480	-2.427	0.604	-3.074	6
2	23.40	22.89	0.51	0.846	0.122	0.009	0.117	13
3	42.50	56.89	-14.39	0.137	-1.459	0.037	-1.527	9
4	46.10	53.12	-7.02	0.316	-0.800	0.033	-0.789	3
5	30.80	25.97	4.83	0.929	1.705	4.220	1.846	1
6	62.50	59.98	2.52	0.255	0.275	0.003	0.266	15
7	38.20	45.75	-7.55	0.338	-0.874	0.043	-0.866	2
8	75.40	62.84	12.56	0.101	1.247	0.019	1.274	10
9	44.80	46.66	-1.86	0.314	-0.211	0.002	-0.204	14
10	34.80	39.68	-4.88	0.272	-0.539	0.012	-0.524	8
11	52.80	58.82	-6.02	0.189	-0.630	0.010	-0.616	4
12	40.20	40.97	- 0.77	0.834	-0.177	0.018	-0.171	12
13	80.00	62.84	17.16	0.101	1.704	0.036	1.844	5
14	50.60	57.54	-6.94	0.176	-0.720	0.012	-0.707	11
15	75.40	62.84	12.56	0.101	1.247	0.019	1.274	16
16	23.40	17.80	5.60	0.735	1.024	0.323	1.026	7
17	55.60	55.26	0.34	0.756	0.064	0.001	0.062	17
18	40.30	37.36	2.94	0.809	0.634	0.189	0.619	18
19	42.70	47.30	-4.60	0.295	-0.516	0.012	-0.502	19
20	54.20	54.82	-0.62	0.350	-0.072	0.000	-0.070	20
21	60.00	59.92	0.083	0.371	0.010	0.000	0.009	21
22	68.00	62.59	5.41	0.197	0.568	0.009	0.554	22
		23	71.60	62.85	8.75	0.101		
			0.869	0.009	0.861	23		

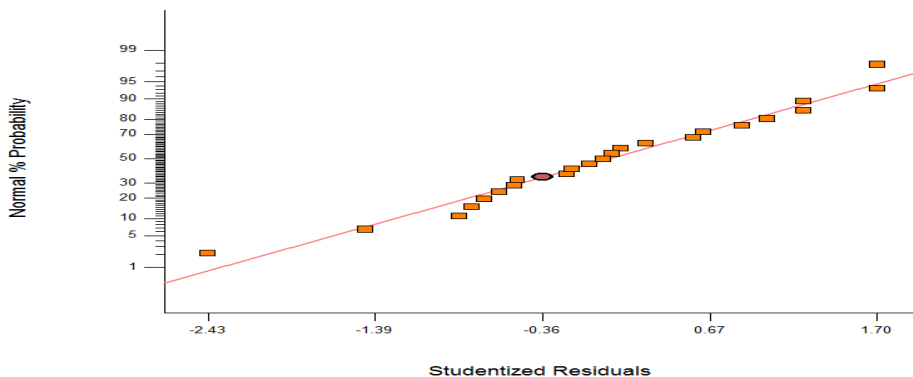


Fig. 4.31: Normal Probability Plot of Residuals for Removal Efficiency APS

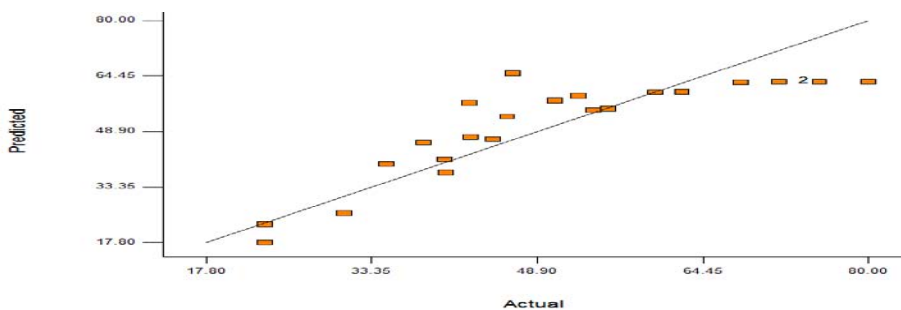


Fig. 4.32: Predicted vs. Actual Values for Removal Efficiency of APS

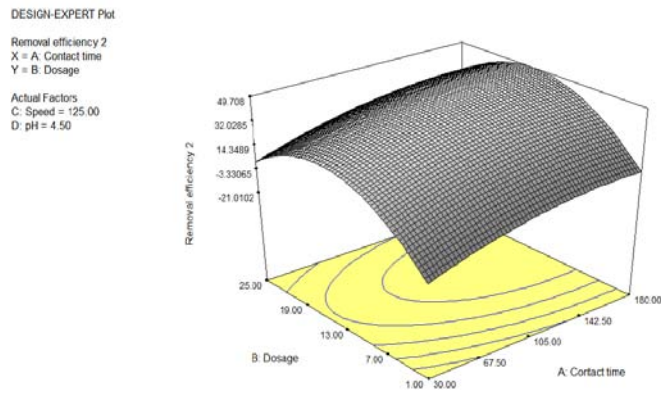


Fig. 4.33: 3D Plot Showing the Effects of Contact Time and Dosage on Removal efficiency of APS

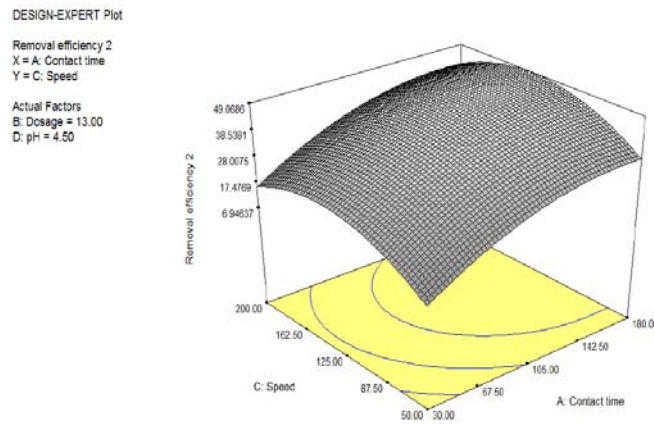


Fig. 4.34: 3D Plot Sowing the Effects of Speed and Contact Time on Removal efficiency of APS

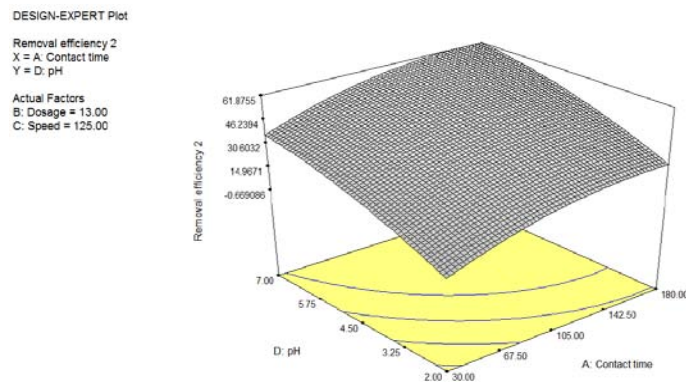


Fig. 4.35: 3D Plot Sowing the Effects of pH and Contact Time on Removal Efficiency of APS

Table 4.19: Table of solutions found for Cr removal using APS

Solutions						
No.	A	B	C	D	E(%)	Desirability
1	168.10	15.141	38.80	7.00	63.3043	0.705 Selected
2	169.35	15.221	37.51	7.00	63.298	0.705
3	168.80	15.301	39.73	7.00	63.2959	0.705
4	166.56	14.911	39.46	7.00	63.2879	0.705
5	170.81	15.011	37.23	7.00	63.2858	0.705
6	172.65	15.041	35.47	7.00	63.2522	0.704
7	175.64	15.001	36.09	7.00	63.2093	0.703

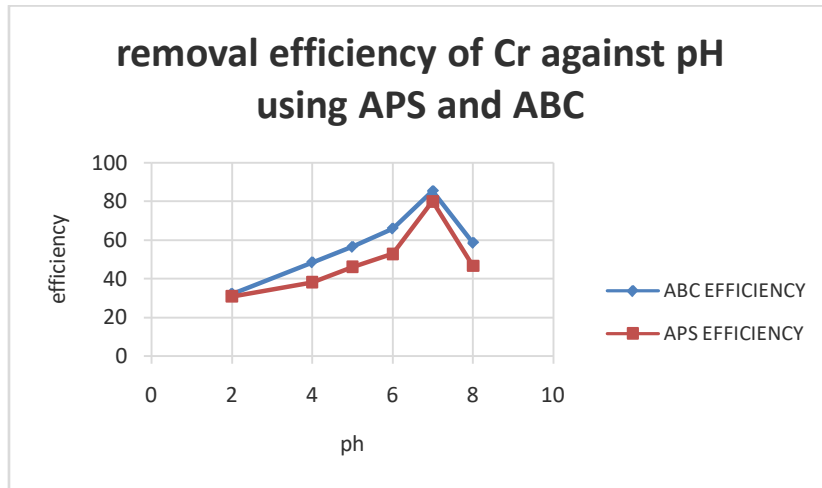


Fig .4.36: Removal efficiency of Cr against pH using APS and ABC

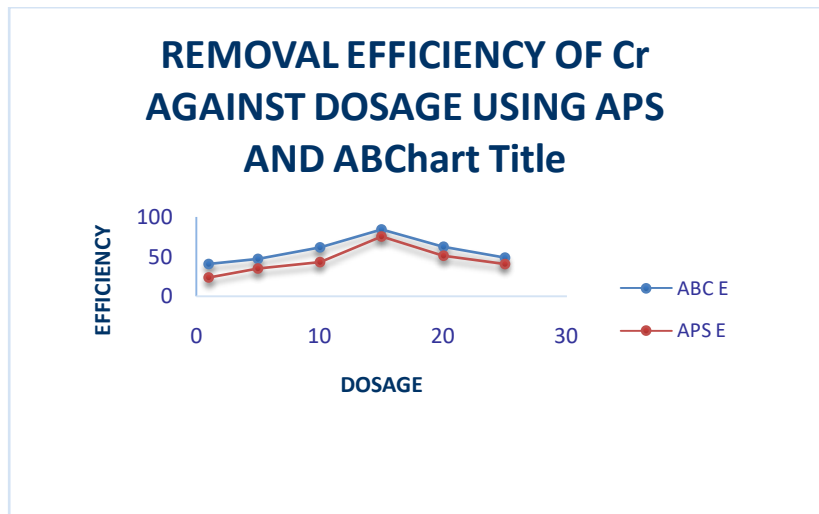


Fig. 4.37: Removal efficiency of Cr against adsorbent dosage using APS and ABC

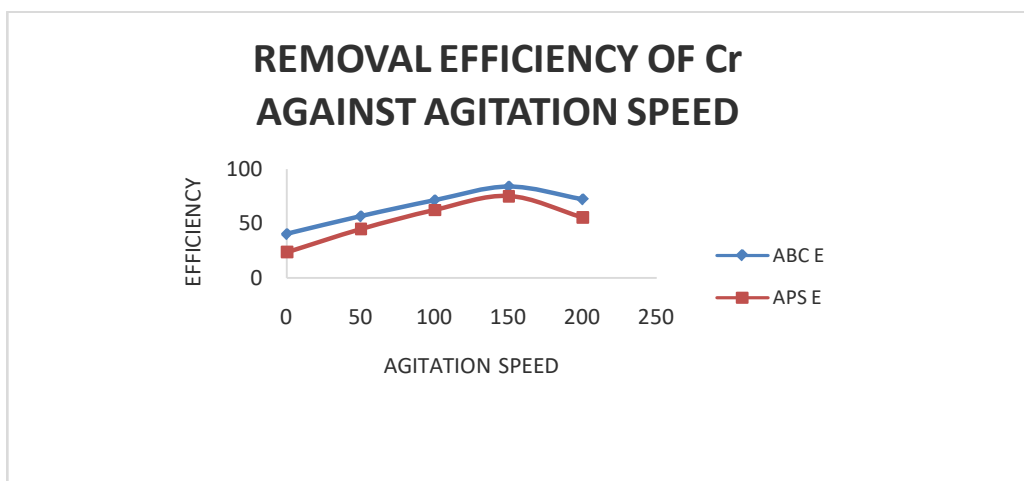


Fig. 4.38: Removal efficiency of Cr against Agitation speed

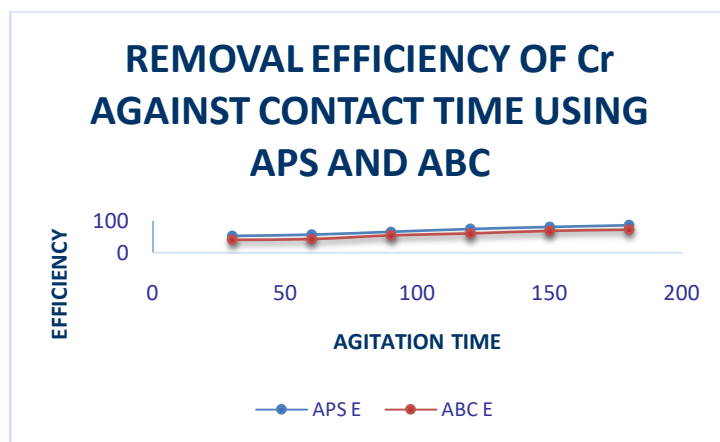


Fig. 4.39: Removal efficiency of Cr against contact time using APS and ABC

V. CONCLUSION

The adsorption of Chromium using Bamboo carbon (ABC) and Periwinkle shell carbon (APS) has been modeled and studied statistically using a pure-quadratic statistical model and the model was adequate for all cases.

Bamboo carbon showed higher removal efficiency than Periwinkle shell carbon in the range of 3-6%.

Dosage and agitation speed had the most significant effect on removal efficiency, with optimum dosage falling in the range of 12.9-16.2 mg/l, while range of optimum agitation speed could be from 138-200 rpm, which was the maximum used.

The maximum removal efficiencies of the carbon samples in heavy metal removal were least for Chromium, higher for Lead, with Cadmium having the highest removal efficiency. These removal efficiencies varied from 63.3-102.83% for APS and from 84-100.7% for ABC.

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APPENDIX

Data for Pseudo first order for Cr(III) ions for ABC and APS

t(min)	30	60	90	120	150
qt (ABC)	0.526	0.564	0.652	0.738	0.841
qt (APS)	0.403	0.427	0.542	0.600	0.754
qe – qt (ABC)	0.317	0.277	0.189	0.103	0.041
qe – qt (APS)	0.351	0.327	0.212	0.154	0.075
Log(qe – qt) (ABC)	-0.502	-0.558	-0.724	-0.987	-1.23
Log (qe – qt) (APS)	-0.455	-0.485	-0.674	-0.812	-1.01

Data for Pseudo second order for Cr (III) ions for ABC and APS

t(min)	30	60	90	120	180
qt (ABC)	0.526	0.564	0.652	0.738	0.841
qt (APS)	0.405	0.427	0.542	0.600	0.754
t/qt (ABC)	57.03	106.38	138.04	162.60	214.03
t/qt (APS)	74.44	140.52	166.05	200.00	238.73

Data for Intraparticle diffusion model for Cr (III) ions for ABC and APS

t(min)	30	60	90	120	180
0.5logt	0.739	0.889	0.977	1.040	1.128
qt(ABC)	0.526	0.564	0.652	0.738	0.841
qt(APS)	0.403	0.427	0.542	0.600	0.754
Log qt(ABC)	-0.279	-0.249	-0.186	-0.132	-0.075
Log qt(APS)	-0.395	-0.370	-0.266	-0.222	-0.123



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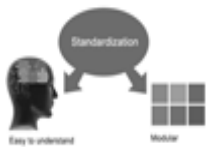
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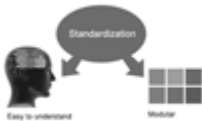


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Color charges: Authors are advised to pay the full cost for the reproduction of their color artwork. Hence, please note that if there is color artwork in your manuscript when it is accepted for publication, we would require you to complete and return a Color Work Agreement form before your paper can be published. Also, you can email your editor to remove the color fee after acceptance of the paper.

TIPS FOR WRITING A GOOD QUALITY ENGINEERING RESEARCH PAPER

Techniques for writing a good quality engineering research paper:

1. Choosing the topic: In most cases, the topic is selected by the interests of the author, but it can also be suggested by the guides. You can have several topics, and then judge which you are most comfortable with. This may be done by asking several questions of yourself, like "Will I be able to carry out a search in this area? Will I find all necessary resources to accomplish the search? Will I be able to find all information in this field area?" If the answer to this type of question is "yes," then you ought to choose that topic. In most cases, you may have to conduct surveys and visit several places. Also, you might have to do a lot of work to find all the rises and falls of the various data on that subject. Sometimes, detailed information plays a vital role, instead of short information. Evaluators are human: The first thing to remember is that evaluators are also human beings. They are not only meant for rejecting a paper. They are here to evaluate your paper. So present your best aspect.

2. Think like evaluators: If you are in confusion or getting demotivated because your paper may not be accepted by the evaluators, then think, and try to evaluate your paper like an evaluator. Try to understand what an evaluator wants in your research paper, and you will automatically have your answer. Make blueprints of paper: The outline is the plan or framework that will help you to arrange your thoughts. It will make your paper logical. But remember that all points of your outline must be related to the topic you have chosen.

3. Ask your guides: If you are having any difficulty with your research, then do not hesitate to share your difficulty with your guide (if you have one). They will surely help you out and resolve your doubts. If you can't clarify what exactly you require for your work, then ask your supervisor to help you with an alternative. He or she might also provide you with a list of essential readings.

4. Use of computer is recommended: As you are doing research in the field of research engineering then this point is quite obvious. Use right software: Always use good quality software packages. If you are not capable of judging good software, then you can lose the quality of your paper unknowingly. There are various programs available to help you which you can get through the internet.

5. Use the internet for help: An excellent start for your paper is using Google. It is a wondrous search engine, where you can have your doubts resolved. You may also read some answers for the frequent question of how to write your research paper or find a model research paper. You can download books from the internet. If you have all the required books, place importance on reading, selecting, and analyzing the specified information. Then sketch out your research paper. Use big pictures: You may use encyclopedias like Wikipedia to get pictures with the best resolution. At Global Journals, you should strictly follow [here](#).



6. Bookmarks are useful: When you read any book or magazine, you generally use bookmarks, right? It is a good habit which helps to not lose your continuity. You should always use bookmarks while searching on the internet also, which will make your search easier.

7. Revise what you wrote: When you write anything, always read it, summarize it, and then finalize it.

8. Make every effort: Make every effort to mention what you are going to write in your paper. That means always have a good start. Try to mention everything in the introduction—what is the need for a particular research paper. Polish your work with good writing skills and always give an evaluator what he wants. Make backups: When you are going to do any important thing like making a research paper, you should always have backup copies of it either on your computer or on paper. This protects you from losing any portion of your important data.

9. Produce good diagrams of your own: Always try to include good charts or diagrams in your paper to improve quality. Using several unnecessary diagrams will degrade the quality of your paper by creating a hodgepodge. So always try to include diagrams which were made by you to improve the readability of your paper. Use of direct quotes: When you do research relevant to literature, history, or current affairs, then use of quotes becomes essential, but if the study is relevant to science, use of quotes is not preferable.

10. Use proper verb tense: Use proper verb tenses in your paper. Use past tense to present those events that have happened. Use present tense to indicate events that are going on. Use future tense to indicate events that will happen in the future. Use of wrong tenses will confuse the evaluator. Avoid sentences that are incomplete.

11. Pick a good study spot: Always try to pick a spot for your research which is quiet. Not every spot is good for studying.

12. Know what you know: Always try to know what you know by making objectives, otherwise you will be confused and unable to achieve your target.

13. Use good grammar: Always use good grammar and words that will have a positive impact on the evaluator; use of good vocabulary does not mean using tough words which the evaluator has to find in a dictionary. Do not fragment sentences. Eliminate one-word sentences. Do not ever use a big word when a smaller one would suffice.

Verbs have to be in agreement with their subjects. In a research paper, do not start sentences with conjunctions or finish them with prepositions. When writing formally, it is advisable to never split an infinitive because someone will (wrongly) complain. Avoid clichés like a disease. Always shun irritating alliteration. Use language which is simple and straightforward. Put together a neat summary.

14. Arrangement of information: Each section of the main body should start with an opening sentence, and there should be a changeover at the end of the section. Give only valid and powerful arguments for your topic. You may also maintain your arguments with records.

15. Never start at the last minute: Always allow enough time for research work. Leaving everything to the last minute will degrade your paper and spoil your work.

16. Multitasking in research is not good: Doing several things at the same time is a bad habit in the case of research activity. Research is an area where everything has a particular time slot. Divide your research work into parts, and do a particular part in a particular time slot.

17. Never copy others' work: Never copy others' work and give it your name because if the evaluator has seen it anywhere, you will be in trouble. Take proper rest and food: No matter how many hours you spend on your research activity, if you are not taking care of your health, then all your efforts will have been in vain. For quality research, take proper rest and food.

18. Go to seminars: Attend seminars if the topic is relevant to your research area. Utilize all your resources.

19. Refresh your mind after intervals: Try to give your mind a rest by listening to soft music or sleeping in intervals. This will also improve your memory. Acquire colleagues: Always try to acquire colleagues. No matter how sharp you are, if you acquire colleagues, they can give you ideas which will be helpful to your research.

20. Think technically: Always think technically. If anything happens, search for its reasons, benefits, and demerits. Think and then print: When you go to print your paper, check that tables are not split, headings are not detached from their descriptions, and page sequence is maintained.



21. Adding unnecessary information: Do not add unnecessary information like "I have used MS Excel to draw graphs." Irrelevant and inappropriate material is superfluous. Foreign terminology and phrases are not apropos. One should never take a broad view. Analogy is like feathers on a snake. Use words properly, regardless of how others use them. Remove quotations. Puns are for kids, not grunt readers. Never oversimplify: When adding material to your research paper, never go for oversimplification; this will definitely irritate the evaluator. Be specific. Never use rhythmic redundancies. Contractions shouldn't be used in a research paper. Comparisons are as terrible as clichés. Give up ampersands, abbreviations, and so on. Remove commas that are not necessary. Parenthetical words should be between brackets or commas. Understatement is always the best way to put forward earth-shaking thoughts. Give a detailed literary review.

22. Report concluded results: Use concluded results. From raw data, filter the results, and then conclude your studies based on measurements and observations taken. An appropriate number of decimal places should be used. Parenthetical remarks are prohibited here. Proofread carefully at the final stage. At the end, give an outline to your arguments. Spot perspectives of further study of the subject. Justify your conclusion at the bottom sufficiently, which will probably include examples.

23. Upon conclusion: Once you have concluded your research, the next most important step is to present your findings. Presentation is extremely important as it is the definite medium through which your research is going to be in print for the rest of the crowd. Care should be taken to categorize your thoughts well and present them in a logical and neat manner. A good quality research paper format is essential because it serves to highlight your research paper and bring to light all necessary aspects of your research.

INFORMAL GUIDELINES OF RESEARCH PAPER WRITING

Key points to remember:

- Submit all work in its final form.
- Write your paper in the form which is presented in the guidelines using the template.
- Please note the criteria peer reviewers will use for grading the final paper.

Final points:

One purpose of organizing a research paper is to let people interpret your efforts selectively. The journal requires the following sections, submitted in the order listed, with each section starting on a new page:

The introduction: This will be compiled from reference matter and reflect the design processes or outline of basis that directed you to make a study. As you carry out the process of study, the method and process section will be constructed like that. The results segment will show related statistics in nearly sequential order and direct reviewers to similar intellectual paths throughout the data that you gathered to carry out your study.

The discussion section:

This will provide understanding of the data and projections as to the implications of the results. The use of good quality references throughout the paper will give the effort trustworthiness by representing an alertness to prior workings.

Writing a research paper is not an easy job, no matter how trouble-free the actual research or concept. Practice, excellent preparation, and controlled record-keeping are the only means to make straightforward progression.

General style:

Specific editorial column necessities for compliance of a manuscript will always take over from directions in these general guidelines.

To make a paper clear: Adhere to recommended page limits.

Mistakes to avoid:

- Insertion of a title at the foot of a page with subsequent text on the next page.
- Separating a table, chart, or figure—confine each to a single page.
- Submitting a manuscript with pages out of sequence.
- In every section of your document, use standard writing style, including articles ("a" and "the").
- Keep paying attention to the topic of the paper.



- Use paragraphs to split each significant point (excluding the abstract).
- Align the primary line of each section.
- Present your points in sound order.
- Use present tense to report well-accepted matters.
- Use past tense to describe specific results.
- Do not use familiar wording; don't address the reviewer directly. Don't use slang or superlatives.
- Avoid use of extra pictures—include only those figures essential to presenting results.

Title page:

Choose a revealing title. It should be short and include the name(s) and address(es) of all authors. It should not have acronyms or abbreviations or exceed two printed lines.

Abstract: This summary should be two hundred words or less. It should clearly and briefly explain the key findings reported in the manuscript and must have precise statistics. It should not have acronyms or abbreviations. It should be logical in itself. Do not cite references at this point.

An abstract is a brief, distinct paragraph summary of finished work or work in development. In a minute or less, a reviewer can be taught the foundation behind the study, common approaches to the problem, relevant results, and significant conclusions or new questions.

Write your summary when your paper is completed because how can you write the summary of anything which is not yet written? Wealth of terminology is very essential in abstract. Use comprehensive sentences, and do not sacrifice readability for brevity; you can maintain it succinctly by phrasing sentences so that they provide more than a lone rationale. The author can at this moment go straight to shortening the outcome. Sum up the study with the subsequent elements in any summary. Try to limit the initial two items to no more than one line each.

Reason for writing the article—theory, overall issue, purpose.

- Fundamental goal.
- To-the-point depiction of the research.
- Consequences, including definite statistics—if the consequences are quantitative in nature, account for this; results of any numerical analysis should be reported. Significant conclusions or questions that emerge from the research.

Approach:

- Single section and succinct.
- An outline of the job done is always written in past tense.
- Concentrate on shortening results—limit background information to a verdict or two.
- Exact spelling, clarity of sentences and phrases, and appropriate reporting of quantities (proper units, important statistics) are just as significant in an abstract as they are anywhere else.

Introduction:

The introduction should "introduce" the manuscript. The reviewer should be presented with sufficient background information to be capable of comprehending and calculating the purpose of your study without having to refer to other works. The basis for the study should be offered. Give the most important references, but avoid making a comprehensive appraisal of the topic. Describe the problem visibly. If the problem is not acknowledged in a logical, reasonable way, the reviewer will give no attention to your results. Speak in common terms about techniques used to explain the problem, if needed, but do not present any particulars about the protocols here.

The following approach can create a valuable beginning:

- Explain the value (significance) of the study.
- Defend the model—why did you employ this particular system or method? What is its compensation? Remark upon its appropriateness from an abstract point of view as well as pointing out sensible reasons for using it.
- Present a justification. State your particular theory(-ies) or aim(s), and describe the logic that led you to choose them.
- Briefly explain the study's tentative purpose and how it meets the declared objectives.



Approach:

Use past tense except for when referring to recognized facts. After all, the manuscript will be submitted after the entire job is done. Sort out your thoughts; manufacture one key point for every section. If you make the four points listed above, you will need at least four paragraphs. Present surrounding information only when it is necessary to support a situation. The reviewer does not desire to read everything you know about a topic. Shape the theory specifically—do not take a broad view.

As always, give awareness to spelling, simplicity, and correctness of sentences and phrases.

Procedures (methods and materials):

This part is supposed to be the easiest to carve if you have good skills. A soundly written procedures segment allows a capable scientist to replicate your results. Present precise information about your supplies. The suppliers and clarity of reagents can be helpful bits of information. Present methods in sequential order, but linked methodologies can be grouped as a segment. Be concise when relating the protocols. Attempt to give the least amount of information that would permit another capable scientist to replicate your outcome, but be cautious that vital information is integrated. The use of subheadings is suggested and ought to be synchronized with the results section.

When a technique is used that has been well-described in another section, mention the specific item describing the way, but draw the basic principle while stating the situation. The purpose is to show all particular resources and broad procedures so that another person may use some or all of the methods in one more study or referee the scientific value of your work. It is not to be a step-by-step report of the whole thing you did, nor is a methods section a set of orders.

Materials:

Materials may be reported in part of a section or else they may be recognized along with your measures.

Methods:

- Report the method and not the particulars of each process that engaged the same methodology.
- Describe the method entirely.
- To be succinct, present methods under headings dedicated to specific dealings or groups of measures.
- Simplify—detail how procedures were completed, not how they were performed on a particular day.
- If well-known procedures were used, account for the procedure by name, possibly with a reference, and that's all.

Approach:

It is embarrassing to use vigorous voice when documenting methods without using first person, which would focus the reviewer's interest on the researcher rather than the job. As a result, when writing up the methods, most authors use third person passive voice.

Use standard style in this and every other part of the paper—avoid familiar lists, and use full sentences.

What to keep away from:

- Resources and methods are not a set of information.
- Skip all descriptive information and surroundings—save it for the argument.
- Leave out information that is immaterial to a third party.

Results:

The principle of a results segment is to present and demonstrate your conclusion. Create this part as entirely objective details of the outcome, and save all understanding for the discussion.

The page length of this segment is set by the sum and types of data to be reported. Use statistics and tables, if suitable, to present consequences most efficiently.

You must clearly differentiate material which would usually be incorporated in a study editorial from any unprocessed data or additional appendix matter that would not be available. In fact, such matters should not be submitted at all except if requested by the instructor.



Content:

- Sum up your conclusions in text and demonstrate them, if suitable, with figures and tables.
- In the manuscript, explain each of your consequences, and point the reader to remarks that are most appropriate.
- Present a background, such as by describing the question that was addressed by creation of an exacting study.
- Explain results of control experiments and give remarks that are not accessible in a prescribed figure or table, if appropriate.
- Examine your data, then prepare the analyzed (transformed) data in the form of a figure (graph), table, or manuscript.

What to stay away from:

- Do not discuss or infer your outcome, report surrounding information, or try to explain anything.
- Do not include raw data or intermediate calculations in a research manuscript.
- Do not present similar data more than once.
- A manuscript should complement any figures or tables, not duplicate information.
- Never confuse figures with tables—there is a difference.

Approach:

As always, use past tense when you submit your results, and put the whole thing in a reasonable order.

Put figures and tables, appropriately numbered, in order at the end of the report.

If you desire, you may place your figures and tables properly within the text of your results section.

Figures and tables:

If you put figures and tables at the end of some details, make certain that they are visibly distinguished from any attached appendix materials, such as raw facts. Whatever the position, each table must be titled, numbered one after the other, and include a heading. All figures and tables must be divided from the text.

Discussion:

The discussion is expected to be the trickiest segment to write. A lot of papers submitted to the journal are discarded based on problems with the discussion. There is no rule for how long an argument should be.

Position your understanding of the outcome visibly to lead the reviewer through your conclusions, and then finish the paper with a summing up of the implications of the study. The purpose here is to offer an understanding of your results and support all of your conclusions, using facts from your research and generally accepted information, if suitable. The implication of results should be fully described.

Infer your data in the conversation in suitable depth. This means that when you clarify an observable fact, you must explain mechanisms that may account for the observation. If your results vary from your prospect, make clear why that may have happened. If your results agree, then explain the theory that the proof supported. It is never suitable to just state that the data approved the prospect, and let it drop at that. Make a decision as to whether each premise is supported or discarded or if you cannot make a conclusion with assurance. Do not just dismiss a study or part of a study as "uncertain."

Research papers are not acknowledged if the work is imperfect. Draw what conclusions you can based upon the results that you have, and take care of the study as a finished work.

- You may propose future guidelines, such as how an experiment might be personalized to accomplish a new idea.
- Give details of all of your remarks as much as possible, focusing on mechanisms.
- Make a decision as to whether the tentative design sufficiently addressed the theory and whether or not it was correctly restricted. Try to present substitute explanations if they are sensible alternatives.
- One piece of research will not counter an overall question, so maintain the large picture in mind. Where do you go next? The best studies unlock new avenues of study. What questions remain?
- Recommendations for detailed papers will offer supplementary suggestions.



Approach:

When you refer to information, differentiate data generated by your own studies from other available information. Present work done by specific persons (including you) in past tense.

Describe generally acknowledged facts and main beliefs in present tense.

THE ADMINISTRATION RULES

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CRITERION FOR GRADING A RESEARCH PAPER (COMPILATION)
BY GLOBAL JOURNALS

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Topics	Grades		
	A-B	C-D	E-F
<i>Abstract</i>	Clear and concise with appropriate content, Correct format. 200 words or below	Unclear summary and no specific data, Incorrect form Above 200 words	No specific data with ambiguous information Above 250 words
<i>Introduction</i>	Containing all background details with clear goal and appropriate details, flow specification, no grammar and spelling mistake, well organized sentence and paragraph, reference cited	Unclear and confusing data, appropriate format, grammar and spelling errors with unorganized matter	Out of place depth and content, hazy format
<i>Methods and Procedures</i>	Clear and to the point with well arranged paragraph, precision and accuracy of facts and figures, well organized subheads	Difficult to comprehend with embarrassed text, too much explanation but completed	Incorrect and unorganized structure with hazy meaning
<i>Result</i>	Well organized, Clear and specific, Correct units with precision, correct data, well structuring of paragraph, no grammar and spelling mistake	Complete and embarrassed text, difficult to comprehend	Irregular format with wrong facts and figures
<i>Discussion</i>	Well organized, meaningful specification, sound conclusion, logical and concise explanation, highly structured paragraph reference cited	Wordy, unclear conclusion, spurious	Conclusion is not cited, unorganized, difficult to comprehend
<i>References</i>	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring



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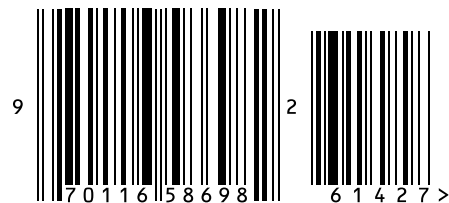


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