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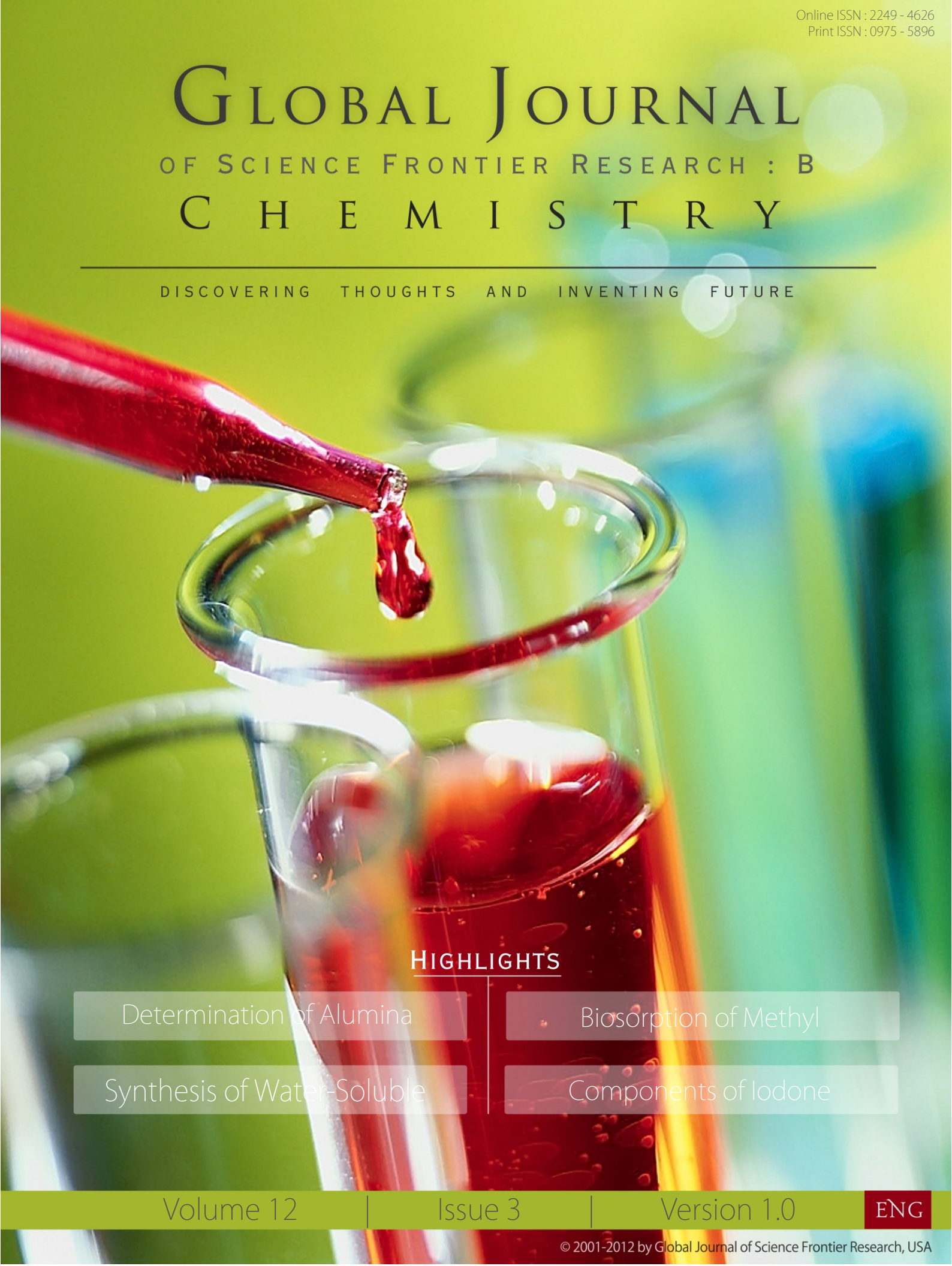

Highlights

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Determination of Alumina Oxide in Bauxites by X-Ray Fluorescence Analysis

By Dragana Keselj , Dragica Lazic , Jelena Penavin-Skundric, Slavica Sladojevic & Ljubica Vasiljevic

Faculty of Technology Zvornik, RS, BiH

Abstract - This study relates to determination of the content of aluminium oxide (%Al2O3) in different types of bauxite by the X-ray fluorescence method (XRF). The samples were prepared in the form of beads by the borax method from bauxite, which had been previously annealed. Standard reference samples of bauxite were used to produce a calibration curve and the calibration curve obtained was with very good coefficient of determination r = 0.9992 and standard deviation S = 0.091. After statistical verification of the method (F-test, reference method and standard sample of bauxite), it was concluded that the method was precise and correct and that there were no systemic errors. In addition to this, by the XRF analysis of different types of bauxite the average value of residuals between percent of Al2O3 determined by the standard method of SRPS B.G8.512 and XRF method was 0.254 with the deviation of 0.191.

Keywords : *bauxite, reference sample, X-ray fluorescence analysis, standard deviation.*

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Determination of Alumina Oxide in Bauxites by X-Ray Fluorescence Analysis

Dragana Keselj [α], Dragica Lazic [σ], Jelena Penavin-Skundric [ρ], Slavica Sladojevic [ω] & Ljubica Vasiljevic [¥]

Abstract - This study relates to determination of the content of aluminium oxide (%Al_2O_3) in different types of bauxite by the X-ray fluorescence method (XRF). The samples were prepared in the form of beads by the borax method from bauxite, which had been previously annealed. Standard reference samples of bauxite were used to produce a calibration curve and the calibration curve obtained was with very good coefficient of determination r = 0.9992 and standard deviation S = 0.091. After statistical verification of the method (F-test, reference method and standard sample of bauxite), it was concluded that the method was precise and correct and that there were no systemic errors. In addition to this, by the XRF analysis of different types of bauxite the average value of residuals between percent of Al_2O_3 determined by the standard method of SRPS B.G8.512 and XRF method was 0.254 with the deviation of 0.191.

Keywords : bauxite, reference sample, X-ray fluorescence analysis, standard deviation.

I. Introduction

Bauxite is the aluminium ore which consists of more than a hundred of different minerals, out of which the most significant ones are the minerals of aluminium, iron, silicon, titanium and calcium. Aluminium is present in bauxite mainly in the form of hydrated oxides: hydrargillite (gibbsite)- $Al_2O_3 \cdot 3H_2O$, boehmite- AlOOH and diaspore- AlOOH. In addition to these minerals, aluminium may be found in bauxite to a lesser extent in the form of corundum (Al_2O_3) and various aluminosilicates, most often in the form of kaolinite- $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$. Silicon in bauxite is present in the form of free or bonded oxide. Minerals of free oxides, which may be found in bauxite, are crystal forms of SiO_2, those being: quartz, quartzite, chalcedony or amorphous SiO_2 - opal. Out of free oxides, SiO_2 may most often be found in the form of quartz mineral. As a bonded oxide, silicon is usually found as the mineral of kaolinite- $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$. Iron is generally found in bauxites as anhydrous and hydrated oxides, those being : hematite - Fe_2O_3, magnetite - Fe_3O_4, hydrated hematite- $Fe_2O_3 \cdot H_2O$, goethite-$HFeO_2$ and limonite-$HFeO_2 \cdot H_2O$. Titanium is most frequently present in bauxites in the form of a free oxide and appears in three allotropic modifications of anatase, rutile and brookite, out of which anatase-TiO_2 is most frequent. Calcium appears in the form of various carbonates, most often as calcite-$CaCO_3$ and dolomite- $MgCO_3 \cdot CaCO_3$. Depending on the mineral form of the present aluminium, as the basic mineral, bauxites are divided into: hydrargillite (gibbsite), boehmite, diaspore and mixed ones (hydrargillite-boehmite and boehmite-diaspore).

Chemical composition of bauxite is presented by the following components: Al_2O_3, SiO_2, Fe_2O_3, TiO_2, CaO and the loss on ignition at 1075°C. Standard method for chemical determination of aluminium in bauxites is conducted based on the SRPS B.G8.512 standard. By this standard bauxites first were decomposed with acids and Al_2O_3 were determinated by potenciometric method.

X-ray fluorescence analysis (XRF) is one of the most significant emission methods, which enables a quick and multielemental analysis in a very short period of time and requires a minimal preparation of the sample. In case some material gets showered with primary high energy X-rays, it will cause expulsion of electrons from some of inner shells (K,L,M) of atoms in that material, which results in formation of electron holes in one or more atomic orbitals closer to the nucleus, by which appropriate atoms get into an excited state. The atom excited in such a way tends to get into a stable state and therefore the holes in orbitals closer to the nucleus get filled up with the electrons from higher orbitals. This transfer is accompanied with the emission of energy in the form of a secondary, i.e. fluorescent X-ray, which is characteristic of the given atom. The substance of the X-ray fluorescent analysis is measuring the intensity of the developed secondary fluorescence radiation. The method is applicable in a wide scope of concentrations. For determining the components with a high concentration in the examined sample, determination is performed with the help of the calibration curve with the standards of the known composition, where concentration of the appropriate component is generally proportional to the emitted fluorescence radiation by the element that was determined. Due to its advantages, first of all because it is quick, non-destructive and less expensive, the X-ray

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fluorescence analysis is nowadays applied in many fields, where it is particularly necessary to point out its application in everyday analyses in the cement industry and metallurgy.

II. Eksperimental Part

According to the research so far (La Tour T.E. 1989, Giles et all, 1995), the best results for determining individual components in the multi-component systems by the X-ray fluorescence method are achieved by dissolving and diluting the sample by some of analytes. When the analysis of rocks is in question, it is customary to prepare the sample by the borax method (Alvarez 1990, Nakayama et all 2007, Hettipathirana et all 2004). Based on this method, borax beads are made so that the examined sample gets destroyed by borax ($Na_2B_4O_7$) or by lithium tetraborate ($Li_2B_4O_7$), with or without oxidizing substance (. Since aluminium is most often present in bauxites as a hydrated oxide, at the high temperatures at which borax palettes are made there will occur its dehydration, so that in preparation of palettes of bauxite of a different type there will occur an error caused by different losses on ignition of different types of bauxite. Therefore, in this study the samples of bauxite, which had been previously crushed up to granulation of less than 200 μm and dried up, were annealed at 1200°C and at the same temperature their loss on ignition was determined. 1 g of the bauxite sample was taken from the sample annealed in that manner and it was mixed with 6 g $Li_2B_4O_7$, then it was melted at the temperature of 1200°C for a half an hour with periodical stirring and was poured out into the platinum moulds. The obtained beads in the platinum moulds were recorded on the apparatus.

For measuring the fluorescence radiation the apparatus used was the spectrometer Philips PW1404, where the conditions of recording, i.e. Channelset for aluminium, was: line- Kα, X-tal – PE, collimator – coarse, detector- FL, kV- 40, mA- 75, angle (°2θ)- 144.8650.

In the experimental part, the certified samples of bauxite were used for making the calibration curve (Table 1.).

The study also used bauxites of various deposits and they were processed in the Factory AD „Birac" Zvornik, those deposits being: Milici (S1.), Posusje (S2.), Potoci-Mrkonjic Grad(S3.), Liskavica-Mrkonjic Grad (S4.), Citluk (S5.), Bosanska Krupa (S6.), India (S7.) and Guinea (S8.) (Table 2.).

Mineralogical characterization was performed for all samples of bauxite by the X-ray diffraction, where a copper tube was used for an X-ray tube and had the following characteristics: Anode of Cu, K-Alpha1 [Å]=1.54060, K-Alpha2 [Å]= 1.54443, K-Beta [Å]=1.39225, K-A2 / K-A1 Ratio=0.50000, Generator Settings 50 mA and 40 kV.

Table 1: Analysis of standard reference samples of bauxite according to the certificate

Comp.	Standard reference samples of bauxite					
	NBS 696	NBS 697	NBS 698	BCS 395	SB1	SB2
Al_2O_3	54.5	45.8	48.2	52.4	55.40	48.6
Fe_2O_3	8.70	20.00	19.6	16.30	24.4	28.2
FeO	-	-	-	-	2.2	3.89
SiO_2	3.79	6.81	0.69	1.24	2.38	7.38
TiO_2	2.64	2.52	2.38	1.93	2.22	3.06
CaO	0.018	0.71	0.62	0.05	1.93	0,39
MgO	0.012	0.18	0.058	0.02	0.14	0.39
Na_2O	0.007	0.036	0.015	-	-	-
SO_3	0.21	10.13	0.22	-	-	-
S	-	-	-	-	0.33	0.028
GZ1075°C	29.9	22.1	27.3	27.8	12.7	11.7
Gibbsite	80	50	75	-	-	-
Boehmite	-	10	-	-	50-60	45-50
Diaspore	-	-	-	-		-
Kaolinite	5	15	-	-	5	To 5
Hematite	-	20	20	-	20-22	28-32
Pyrite	10	-	-	-	0,5-1	0-4
Goethite	-	-	-	-	-	-
Anatase	5	5	5	-	-	-
Calcite	-	-	-	-	3-4	1-2

Table 2 : Chemical analysis of different types of bauxites

Com.	Deposits of bauxite							
	S1.	S2.	S3.	S4.	S5.	S6.	S7.	S8.
Al_2O_3	51.22	54.59	45.56	56.20	49.61	48.16	51.54	59.00
Fe_2O_3	25.97	20.69	20.00	20.56	19.50	16.16	18.31	6.70
SiO_2	7.93	1.36	6.48	1.52	5.13	3.78	6.18	1.41
TiO_2	2.58	2.87	2.11	2.60	2.69	2.44	2.37	3.77
CaO	0.16	0.58	1.88	4.00	4.50	1.95	4.63	0.28
GZ 1075 °C	11.35	19.90	23.22	14.11	17.70	26.79	15.95	28.64
GZ 1200 °C	11.35	20.04	23.22	14.11	17.70	27.68	15.95	28.64

III. Results and Discussion

Standard reference samples of bauxite, according to the certificate, have a known mineralogical composition (Table 1). The standard sample of bauxite BCS 395 in the certificate does not contain the information on the mineralogical composition, but it can be seen based on the obtained diffractogram that aluminium is present as gibbsite and in very small traces as boehmite and kaolinite (Picture 1.). Bauxites NBS 696 and NBS 698 are of hydragillite type, NBS 697 and BCS 395 are of hydragillite-

boehmite type, SB2 is of boehmite type and SB1 is of boehmite-diaspore type.

Based on X-ray diffraction analysis of the samples of bauxite from different locations (Pictures 2 - 9), it can be noted that the samples of bauxites S7 and S8 are of gibbsite type, S1 and S6 are of boehmite type and thesamples of bauxites S2, S3, S4 and S5 are of hydragillite-boehmite type. The chemical analysis for the samples was performed based on the standard SRPS B.G8.512 (Table 2.).

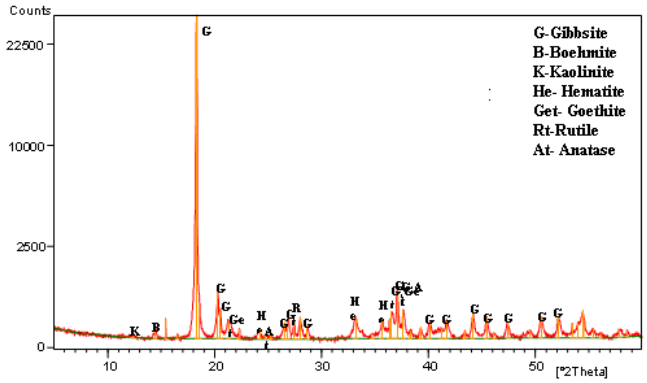

Fig.1 : Diffractogram of the standard sample BCS 395.

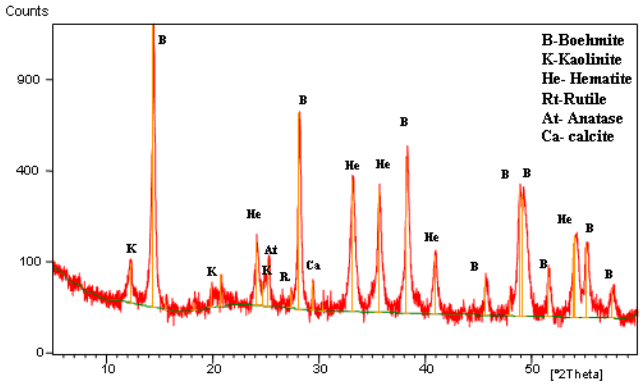

Fig.2 : Diffractogram of the bauxite sample S1.

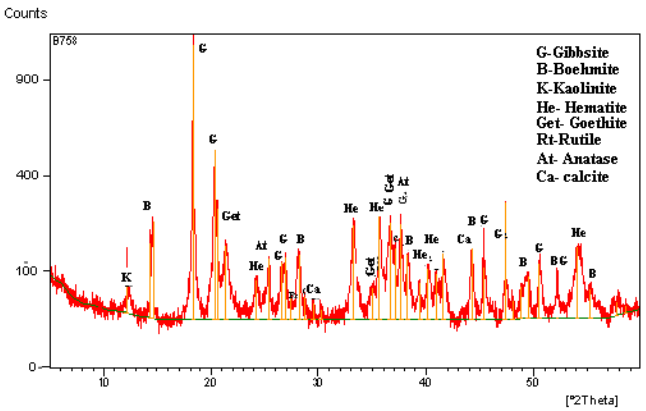

Fig.3 : Diffractogram of the bauxite sample S2.

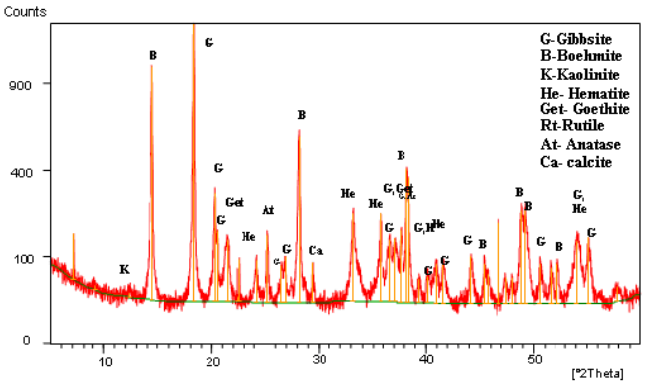

Fig.4 : Diffractogram of the bauxite sample S3 .

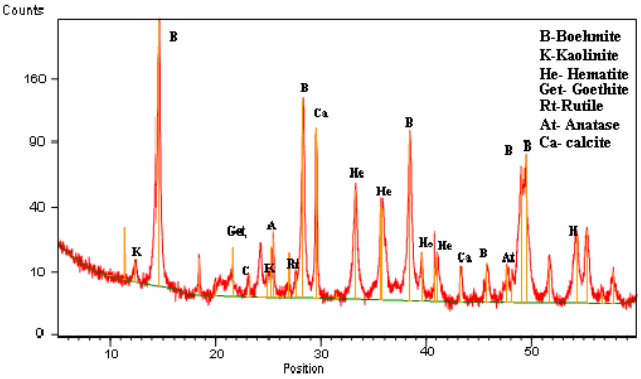

Fig.5 : Diffractogram of the bauxite sample S4 .

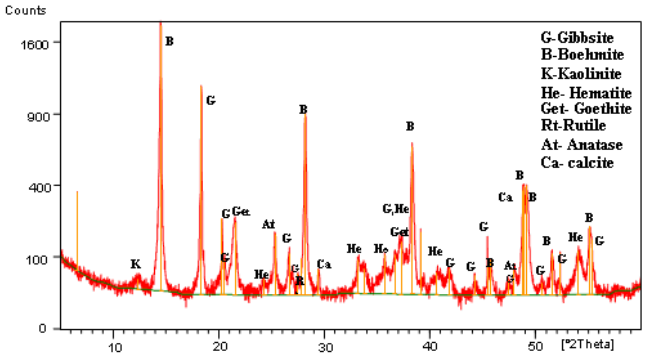

Fig.6 : Diffractogram of the bauxite sample S5 .

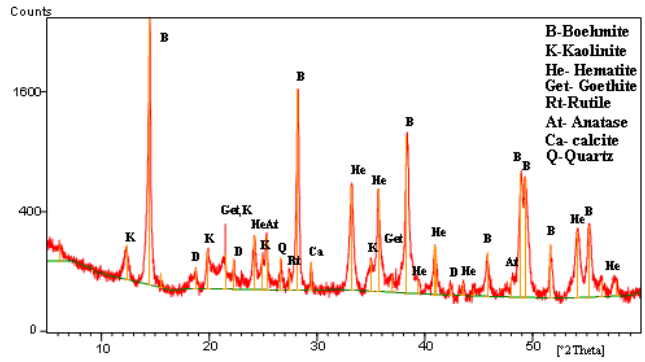

Fig.7 : Diffractogram of the bauxite sample S6 .

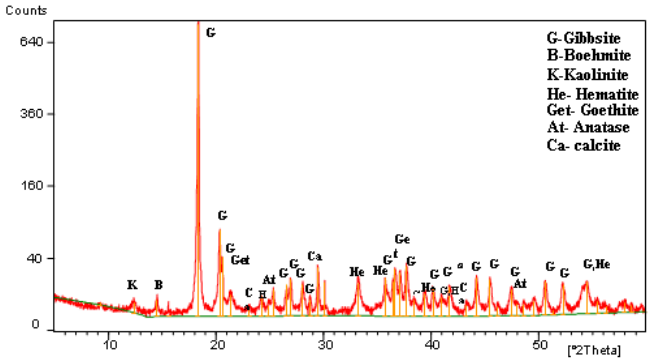

Fig.8 : Diffractogram of the bauxite sample S7.

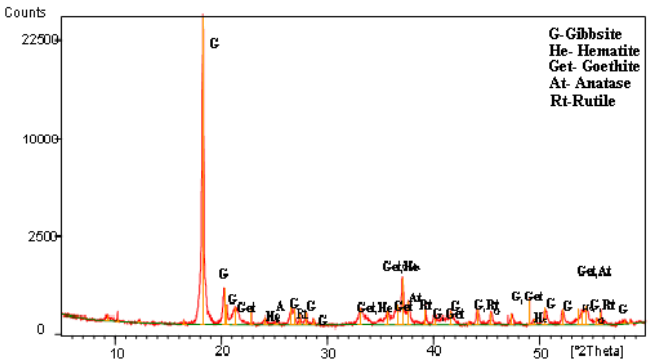

Fig. 9 : Diffractogram of the bauxite sample S8 .

Content of Al_2O_3 taken in the calibration curve (Picture 10.) is calculated for the absolutely annealed sample.

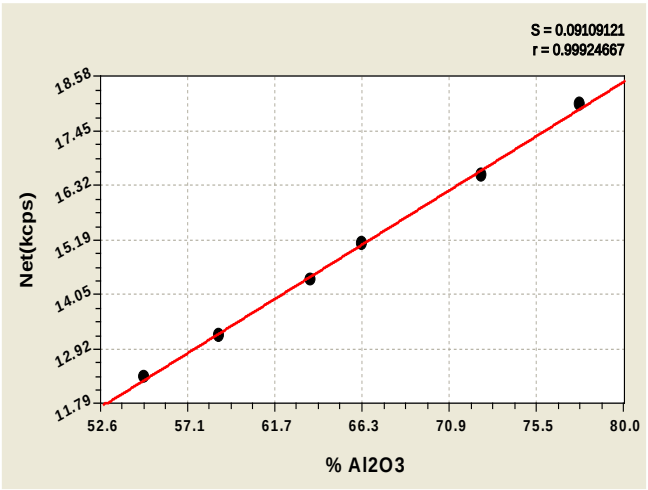

Fig. 10 : Calibration curve .

Based on the calibration curve we got the equation for calculating concentration of aluminium in the annealed bauxite:

$$\%Al_2O_{3\ annealed} = 4.0714689 * INT + 4.8320482 \quad (1)$$

Where INT- masured intensity (kcps)

And the true content of Al_2O_3 in bauxite is further calculated based on the loss on ignition determined at 1200°C ($\mathbf{GZ_{1200}}$) based on the formula:

$$\%Al_2O_3 = \%Al_2O_{3\ annealed} * (100 - GZ_{1200})/100 \quad (2)$$

In accordance with the calibration curve, the samples of bauxite in different deposits were also recorded and the content of Al_2O_3 was determined (Table 3.).

Table 3 : Content of Al_2O_3 in the bauxites of different locations, which was determined by the standard and XRF method

Sample	Content of Al_2O_3 in bauxites, %		Residual
	Standard method	XRF method	
S1.	51.22	51.43	0.21
S2.	54.59	54.39	0.20
S3.	45.56	45.04	0.52
S4.	56.20	56.51	0.31
S5.	49.41	49.95	0.54
S6.	48.16	48.09	0.07
S7.	51.54	51.69	0.15
S8.	59.80	59.83	0.03
Average	-	-	0.254
STDEV	-	-	0.191

Within the framework of the experimental part, ten different beads were recorded for the same sample (Table 4.), as well as one bead for ten times (Table 5.).

Table 4 : Content of Al_2O_3 for ten different beads of the bauxite sample S6

Bead	Net (kcps)	$\%Al_2O_3$ annealed	$\%Al_2O_3$
1.	13.9394	61.586	51.76
2.	14.0227	61.925	52.05
3.	13.8296	61.139	51.39
4.	13.9868	61.779	51.95
5.	13.9333	61.561	51.38
6.	13.9333	61.416	51.62
7.	13.8670	61.291	51.51
8.	13.8952	61.406	51.61
9.	13.9439	61.604	51.78
10.	13.8753	61.325	51.54
Min.	13.8296	61.139	51.54
Max.	14.0227	61.925	52.05
average	13.9227	61.503	51.69
STDEV	0.0576	0.236	0.224

Table 5: Content of Al_2O_3 of one bead of the bauxite sample S6 determined ten times by the XRF method

R.B.Measures	Net (ccps)	$\%Al_2O_3$ annealed	$\%Al_2O_3$
1.	13.9723	61.720	51.88
2.	13.8056	61.041	51.30
3.	13.9210	61.511	51.69
4.	13.9672	61.699	51.86
5.	13.9515	61.635	51.80
6.	13.9296	61.546	51.73
7.	13.9645	61.688	51.85

8.	13.9127	61.477	51.67
9.	13.8498	61.221	51.46
10.	13.9127	61.477	51.67
Min.	13.8056	61.041	51.30
Max.	13.9723	61.720	51.88
Average	13.9187	61.501	51.69
STDEV	0.0539	0.219	0.224

Table 6 : Content of Al_2O_3 of the bauxite sample S6 determined by the standard method of SRPS B.G8.512 for ten samples

R.B.analyses	%Al_2O_3
1.	51.50
2.	51.92
3.	51.84
4.	51.61
5.	51.13
6.	51.73
7.	51.23
8.	51.71
9.	51.19
10.	51.53
Min.	51.13
Max.	51.92
Average	51.54
STDEV	0.277

For the verification of correctness of the new non-standard XRF- method F-test was performed, where a zero hypothesis was tested, by which the variants of the standard method for determination of % Al_2O_3 in bauxites SRPS B.G8.512 and non-standard one are equal.

$$F_{9,9} = \frac{S_1^2}{S_2^2} = \frac{0{,}277^2}{0{,}236^2} = 1{,}378 \qquad (3)$$

The critical value is F_{tab}= 3,179 at α =0.05, which means that the zero hypothesis on the equality of variants was confirmed. The existing differences are considered to be a cause of accidental errors.

Verification of correctness of the XRF method for determination of % Al_2O_3 in bauxites was performed by the use of the standard sample of bauxite B697 (Table 6.).

Table 6 : Chemical analysis of the standard sample of bauxite B697 by the XRF-method

Number	%Al_2O_3
1.	59.034
2.	58.672
3.	58.532
4.	58.715
5.	59.043
6.	58.909
7.	58.446
8.	58.713
9.	58.867
10.	58.404
Min.	58.404
Max.	59.043
Average	58.7335
STDEV	0.229

For the examined XRF method we got:

$$|t| = \left|\frac{\mu - \bar{x}}{s}\right| \cdot \sqrt{n} = 0.822 \qquad (4)$$

where $|t_{tab}| = 2{,}262$ at α=0.05, which means that the XRF method does not have any systemic errors. For the purpose of verifying the XRF method by the reference method of SRPS B.G8.512, the following values were calculated:

$$Sp = \sqrt{\frac{(n_1 - 1)S_1^2 + (n_2 - 1)S_2^2}{n_1 + n_2 - 2}} \sqrt{\left(\frac{1}{n_1} + \frac{1}{n_2}\right)} \qquad (5)$$

$$Sp = 0{,}115$$

$$t = \frac{\bar{x}_1 - \bar{x}_2}{Sp} = \frac{51.693 - 51.539}{0.115} = 1.339 \qquad (6)$$

where t_{tab}= 2.101, so one may conclude that there is no difference between the mean values with the risk of 5 %, i.e. the XRF method does not have any systemic errors.

IV. Conclusion

The calibration curve obtained by recording the beads of standard reference samples of bauxite, which were obtained from the previously annealed samples and then melted with $Li_2B_4O_7$, provided the calibration curve with a very good coefficient of determination r = 0.9992 and standard error S = 0.091. The average value of residuals between % Al_2O_3 determined by the standard method SRPS B.G8.512 and XRF method in different types of bauxite, was 0.254 with the deviation of 0.191. By the verification of correctness of the XRF method for determination of % Al_2O_3 in the bauxites of different types by the reference method and standard sample of bauxite NBS 697, one may conclude that the method does not have any systemic errors. Based on the conducted F-test, we may note that the variants of the standard and XRF method are equal. We may also notice that the standard deviation obtained by recording ten different beads of the „Bosanska Krupa“ sample and standard deviation obtained by recording one bead for ten times are the same. The beads prepared by the

borax method could at the same time serve for determining other components in bauxite. This type of preparation of samples for analysis has the advantage compared to the classical analysis, since the method is non-destructive, a lot fewer chemicals and time is necessary, which gives an advantage to this method.

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Kinetic Studies on Biosorption of Methyl Violet Dye Using Blue Green Algae

By J.Krishnaveni & Dr.N.Renugadevi
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Abstract - In the present study, the parameters, temperature, adsorbent dose, contact time, adsorbent size and agitation speed were optimized for Methyl violet removal from aqueous medium by using response surface methodology (RSM). The optimum conditions for maximum removal of Methyl Violet from an aqueous solution of 100 mg/L were found as follows: room temperature (33 degrees C), adsorbent dose (500mg), contact time (180 min), adsorbent size (250 meshes) and agitation speed (200 rpm). Adsorption kinetic data were tested using Intra-particle diffusion model and Elovich's equation. Kinetic studies showed that the adsorption follows first order reaction. Studies revealed that the intraparticle diffusion plays an important role in the mechanism of dye adsorption.

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Kinetic Studies on Biosorption of Methyl Violet Dye Using Blue Green Algae

J.Krishnaveni [α] & Dr.N.Renugadevi [σ]

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I. Introduction

In recent years, considerable attention has been focused on the removal of dye from aqueous solution using adsorbents derived from low cost materials. Several adsorbents, such as sawdust, silica and iron oxide[1], wheat shell [2], bagasse fly ash [3], fly ash [4], spent activated clay [5] and modified goethite[6] have been used for the treatment of effluents at the solid – liquid interface. In the present investigation Blue Green Algae (BGA) has been used as adsorbent for the removal of Methyl Violet dye. The aim of the present work is to explore the possibility of utilizing BGA for the adsorption of Methyl Violet dye from industrial dye effluents. The kinetics of dye adsorption on adsorbent was analysed by various kinetic models.

II. Methods and Materials

a) Adsorbent

Algae were collected from the pond water, Coimbatore, Tamilnadu, India. It was washed with distilled water several times. The clean algae were dried at room temperature for 30 days. The dried algae were grinded and sieved was labeled as BGA and used for batch mode adsorption experiments.

b) Chemicals

Methyl Violet dye used in this study were of commercial grade. Stock solution of dye was prepared by dissolving accurately weighed amount of Methyl Violet dye in 1000ml distilled water. All experimental solution was prepared by diluting the stock solution to the required concentration. The pH of each experimental solution was adjusted to the required initial pH value using 1N HCl or 1N NaOH before mixing the adsorbent. The absorbance of the dye solution before and after agitation was noted with colorimeter.

c) Response Surface Methodology

The effect of various parameters on the removal of Methyl Violet dye onto the response surface BGA was studied; batch adsorption experiments were conducted at room temperature. For each experiment, 100ml of initial concentration dye solution at pH 6.0 were taken in 250ml Erlenmeyer flask. 500mg of BGA adsorbent is added and was shaken at a constant agitation speed (200 rpm). The supernatant was analysed and the effect of adsorbent dose on the removal of dye was measured with different amounts, different pH and various concentration by contacting time (10, 20, 30, 40, 50, 60, 90, 120, 150 and 180 minutes) till attained equillibrium. The optimum conditions for maximum removal of Methyl Violet from an aqueous solution of 100 mg/L were determined as follows: room temperature (33 degrees C), adsorbent dose (500mg), contact time (180 min), adsorbent size (250 mesh), pH 6.0 and agitation speed (200 rpm).

III. Results and Discussion

Analysis of adsorption data is important for developing kinetic equation that can be used for design purposes. By the above said batch experiments kinetic models have been used to investigate the mechanism of adsorption and potential rate controlling steps, which is helpful for selecting optimum operating conditions for the full- scale batch process[7].

a) Intra-particle diffusion study

The most commonly used technique for identifying the mechanism involved in the adsorption process is by using intra-particle diffusion model as [8,9]

$$q = K_P \sqrt{t}$$

where K_P is the intra-particle diffusion rate constant. If intra-particle diffusion occurs, then q against $\sqrt{t}$ will be linear and the line will pass through the origin if the intra-particle diffusion was the only rate limiting parameter controlling the process. Otherwise, some other mechanism is also involved. Figure: 1 represents

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Intra-particle plot for Methyl Violet onto BGA for different dye concentrations. The figure shows two linear portions, [10, 11] the first part of curve is attributed to boundary layer diffusion while, the final linear parts indicated effect of intra-particle diffusion.

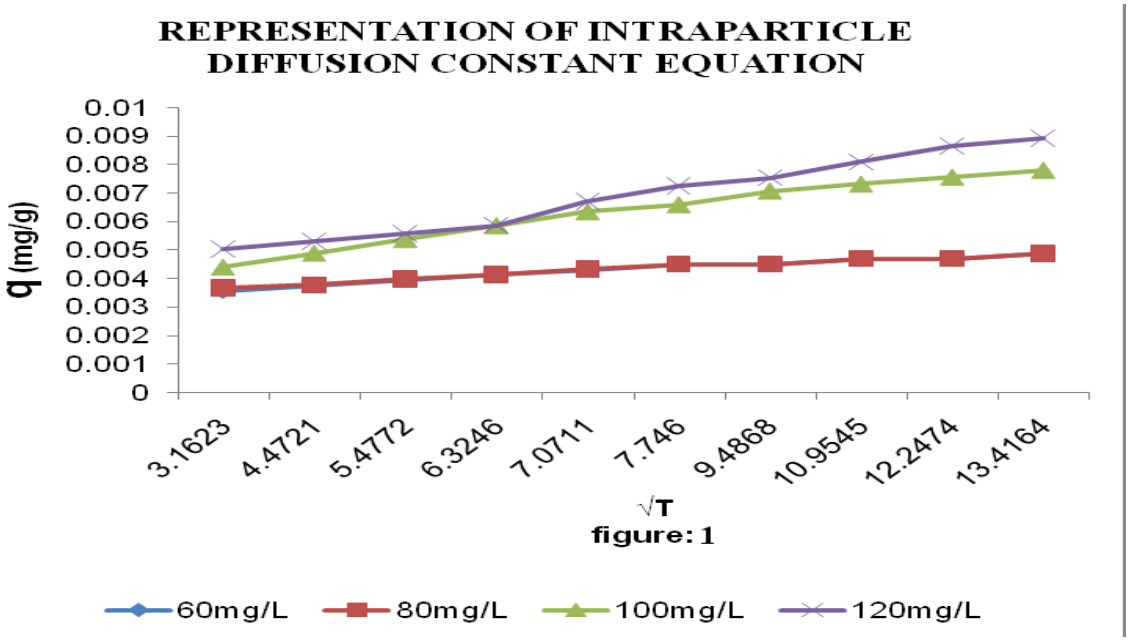

figure: 1

Values of $R^2 = 0.954 - 0.986$ give an idea about the successfulness of the process. The increase of Kp with the increase of MV dye initial concentration shows the thickness of the boundary layer and the constant diffusion of the dye onto BGA . The diffusion rateparameters were shown in Table: 1. The data's indicated that Intra-particle diffusion controls the adsorption rate. Simultaneously, external mass transfer resistance cannot be neglected although this resistance is only significant for the initial period of time [12].

Table 1 : The parameters and correlation coefficients for the removal MV dye on BGA

Concentration of dye solution mg/L	Intraparticle diffusion rate constant		Elovich rateconstant	
	K_p x 10^{-3}	R^2	Desorption constant [β] x 10^3	R^2
60	1.3	0.954	2.174	0.981
80	1.23	0.957	2.304	0.975
100	3.6	0.976	0.762	0.991
120	4.1	0.986	0.673	0.968

b) Elovich's equation

Elovich's equation [13] is given as: $dq_t/dt = \alpha \exp(-\beta q_t)$ Where q_t is the amount of dye adsorbed at time t, α is the initial adsorption rate (mg/g min) and β is the desorption constant (g/mg). After integration and applying boundary conditions, $t = 0$ to t and $q = 0$ to q_e; after integration the above equation becomes: $q_t = \beta \ln(\alpha\beta) + \ln t$

Values of desorption rate constant (β) for the dye adsorption onto BGA were determined from the linear relation of straight line plot of ln t against q_t shown in figure: 2 the data were fitted with a high correlation coefficient (Table: 1) for the removal of dye onto BGA ($R^2 = 0.968 - 0.991$). This shows that the film diffusion is not the only rate controlling parameter. It concluded that the film and pores diffusion were carried out on the surface of BGA adsorbent.

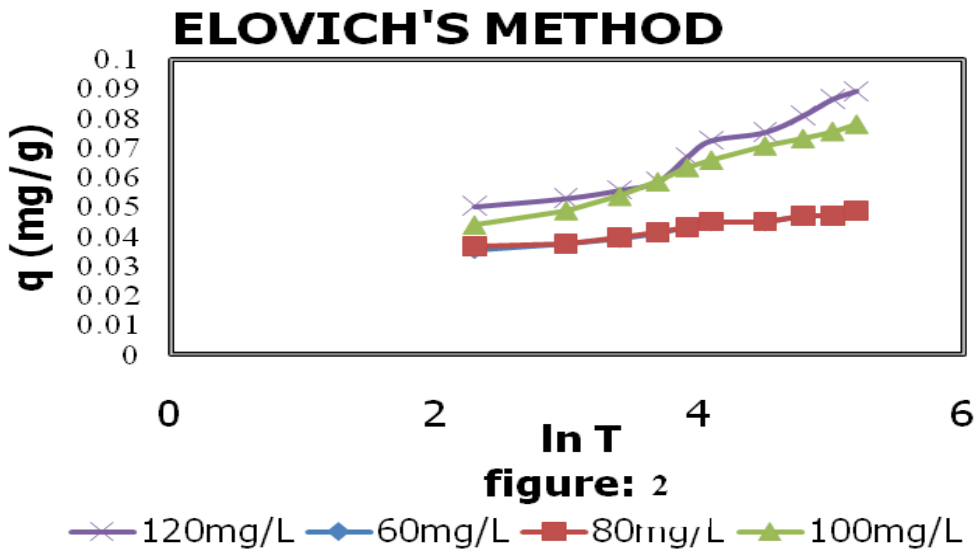

figure: 2

IV. Conclusion

Adsorption of MV onto BGA was best fitted by the first order model confirmed by kinetic models. Mechanism of adsorption is probably a combination of external mass transfer and intra-particle diffusion. A comparison of these values with the one obtained in this study shows that Blue Green Algae used in this research exhibited a higher capacity for MV adsorption from aqueous solutions. Using waste biomass for preparing new biosorbents is particularly advantageous. Blue Green Algae are recognized as a promising class of low-cost adsorbents for the removal of colour from aqueous waste solutions. The application of the adsorption of Methyl Violet dye by using BGA adsorbent will proved its efficiency in wastewater treatment applications.

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Synthesis of Water-Soluble Single-Walled Nanotubes by Functionalization via Esterification

By Javad Azizian, Mahdieh Entezari, Shahab Zomorodbakhsh & Abolghasem Shameli

Islamic Azad University, Tehran, Iran

Abstract - Water soluble compoundes were attached to single-walled carbon nanotubes (SWNTs) to form watersoluble nano dyes. functionalized SWNTs were then characterized by Fourier Transform Infrared spectroscopy (FT-IR), Raman spectroscopy, scanning electron microscopy (SEM) and UV analysis. The product can be dissolved in water and High-resolution transmission electron microscope images showed that the SWNTs were efficiently functionalized, thus the p-stacking interaction between aromatic rings and COOH of SWNTs was considered responsible for the high solubility and High transmission electron in singlewall nanotubes.

Keywords : *Functionalized CNTs, Singlewalled carbon nanotubes, Water soluble compoundes.*

GJSFR-B Classification : *FOR Code: 030605*

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Synthesis of Water-Soluble Single-Walled Nanotubes by Functionalization via Esterification

Javad Azizian [α], Mahdieh Entezari [σ], Shahab Zomorodbakhsh [ρ] & Abolghasem Shameli [ω]

Abstract - Water soluble compoundes were attached to single-walled carbon nanotubes (SWNTs) to form water-soluble nano dyes. functionalized SWNTs were then characterized by Fourier Transform Infrared spectroscopy (FT-IR), Raman spectroscopy, scanning electron microscopy (SEM) and UV analysis. The product can be dissolved in water and High-resolution transmission electron microscope images showed that the SWNTs were efficiently functionalized, thus the p-stacking interaction between aromatic rings and COOH of SWNTs was considered responsible for the high solubility and High transmission electron in singlewall nanotubes.

Keywords : *Functionalized CNTs, Singlewalled carbon nanotubes, Water soluble compoundes.*

I. Introduction

The discovery of carbon nanotubes (CN) and the prospect of developing novel carbon-based nanomaterials has excited worldwide interest among researchers [1, 2]. Single-walled carbon nanotubes (SWNTs) have drawn much attention because of their unique structural, electronic, mechanical and optical properties and the potential applications in nanotechnologies [3]. Organic dyes and pigmentes have a group as their chromophore such as N=N, N=O or SO_3H [4]. The synthesis of water-soluble carbon nanotubes is an important topic because such materials have potential applications in water base systems such as polymers [5-15] crown ethers [16] glucosamines [17] and diazo dyes [18]. Like halogenation and nitration, sulfonation is of the greatest importance in dye manufacture. Most of the water soluble dyes owe their solubility to the presence of sulfonic acid groups. In this paper we present a simple route for sulfonation of organic compoundes[19], and then the products were successfully attached to SWNT–COOH via esterification method.

II. Experimental

All reagents and solvents were obtained from Merck Chemical Inc. and SWNT–COOH (90% purity, 1 – 2 nm) were purchased and used as received. The FT-IR spectrum was recorded using KBr tablets on a Nexus 870 FT- IR spectrometer (Thermo Nicolet, Madison, WI). FT-Raman spectra were recorded on 960 ES spectrometer (Thermo Nicolet), UV-visible spectra were recorded on a UV-Visible spec-trometer (GBC Cintra 20, Victoria, Australia), HNMR spectrum was recorded on Bruker DRX-300 Avance spectrometer at solution in $CDCl_3$ using TMS as internal standard. SEM was used to study the morphology of the SWNTs. SEM measurement was carried out on the XL30 electron microscope (Philips, Amsterdam, Netherlands).

a) Preparation of (E)-4-(2-phenyldiazenyl)-3-hydroxy - naphthalene-2-sulfonic acid

Azo salt 2 was prepared by adding HCl /$NaNO_2$ to aniline 1 at 0 °C. This salt was coupled to β-naphthol 3 and produced azo compound 4, The product and concentrated sulfuric acid heated at 70°C for three hours. Boiling water bath replaced by an ice bath. Then the mixture filtered and washed with alcohol to produce compound 5 [19, 21].

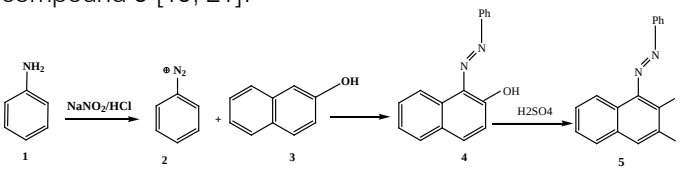

Figure 1 : Synthesis route of (E)-4-(2-phenyldiazenyl)-3-hydroxynaphthalene-2-sulfonic acid

Redish: Orange, power: (85%), mp=120-122^{0}C (decomp), IR(KBr,cm^{-1}), 3400 broad (OH, SO_3H), 2820 (C-H), 1571-1390 (NO_2), 1623 (N=N), 1180 (S=O),740 (S-O). ^{1}HNMR(300MHz,$CDCl_3$) δ: 6.44-8.96 (m,H Aromatic), 9.12(1H, S, OH), 10.83(1H, S, OH), 13.16(1H, S, OH).

b) Preparation of 3–hydroxy –4 –nitrosonaphthalene–2–sulfonic acid

A mixture of 1.5 gr of α-nitroso-β-naphthol [21] and 1.5 mL of concentrated sulfuric acid heated at 70°C for three hours. Boiling water bath replaced by an ice bath. Then the mixture filtered and washed with water. The product was obtained as brown powder (62%), mp =225-227°C. IR (KBr) 3200 (SO_3H), 3174 (OH), 1644 (C=C), 1480 (NO), 1214 (S=O) cm^{-1}. ^{1}HNMR (500.1 MHz, $CDCl_3$) δ: 7.4-8.1(m, Ar), 8.73,8.70 (2H, 2 OH).

c) Preparation of SWNT–(E)-4-(2-phenyldiazenyl)-3-hydroxynaphthalene-2-sulfonic acid and SWNT-3–hydroxy –4 –nitrosonaphthalene–2–sulfonic acid

30 mg of SWNT–COOH were sonicated in 30 mL of N,N-dimethyl formamide (DMF) for 35 minutes to give a homogeneous suspension. Compounds 4 and 6 were added to the SWNT suspension at 0°C. Any

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mixture was stirred at 0°C for two hours and then triethylamine and HCl were added to the mixture. The reaction mixture was kept at room temperature for 7 days (Figure 2). Finally, the final products 7 and 8 were filtered and washed thoroughly with DMF and acetone. Subsequently, the black solids were vacuum-dried at room temperature for 2 hours.

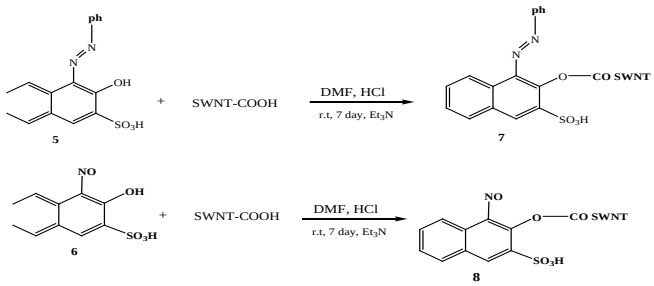

Figure 2 : Synthesis route of modified SWNT-COOH via esterification method

III Results and Discussion

In spectrum FT-IR SWNT-COOH, the band at around 1637 cm^{-1} corresponds to the stretching mode of the C = C double bond that forms the framework of the carbon nanotube sidewall [22]. The peak at 1715 and 3442 cm^{-1} apparently corresponds to the stretching modes of the carboxylic acid groups [23]. The two bands at around 2800–2900 cm^{-1} which are seen in tow spectrum are attributed to the CH stretching of SWNT–COOH defects. In spectrum FT-IR SWNT–(E)-4-(2-phenyldiazenyl)-3-hydroxynaphthalene-2-sulfonic acid, the peak at 1739 cm^{-1} can be attributed to the C=O stretch of the ester. The peaks observed at 1557 and 1353 cm^{-1} are corresponds to the NO_2 group, while the peak at 1280 cm^{-1} corresponds to the S=O in SO_3H group, and the peak at 1118 cm−1 arises from the C–O stretch of the ester group. The band at around 1700 cm^{-1} apparently corresponds to the stretching modes of N=N group [24]. Many of these vibrational modes have been reported previously for functionalized SWCNTs [25]. In spectrum 3, the peak at 3363 cm^{-1} can be assigned to CH stretching of aromatic rings, carbonyl peak in the spectrum 3 shift to 1716 cm^{-1} is a result of ester linkage formation. The band at around 1111 cm^{-1} corresponds to the C-O stretching mode in esters, the peak at 1436 cm^{-1}corresponds to the , N=O the peak at 1183 cm^{-1} corresponds to the S=O in SO_3H group. The peaks at 602 and 800 cm^{-1} are bands originating from the aromatic rings (see the supporting information) [26].

Raman spectra offer useful information concerning the slightly structural changes of SWNTs, especially the changes owing to significant sidewall modification. the characteristic peaks of SWNTs, tangential modes, namely the diameter dependent radial breathing mode (R band) at 210 cm^{-1} depending on the diameter of nanotubes, the D band at around 1330 cm^{-1} and the G band at around 1500 cm^{-1} slightly changed. In this research for SWNT–(E)-4-(2-phenyldiazenyl)-3-hydroxynaphthalene-2-sulfonic acid and SWNT-3–hydroxy –4 –nitrosonaphthalene–2–sulfonic acid, we observed the radial breathing modes were suppressed and shifted to 214 and 219 cm^{-1} by the introduction of the SWNT–(E)-4-(2-phenyldiazenyl)-3-hydroxynaphthalene-2-sulfonic acid and SWNT-3–hydroxy –4 –nitrosonaphthalene–2–sulfonic acid respectively and an increase in the ratio of intensities ID/IG, from 0.65 to 1.12 and from 0.65 to 1 respectively . This indicates an increased disorder of the graphitic structure of the modified nanotubes, which shows that the nanotubes were modified(see the supporting information) [27-34].

More direct evidence for the functionalization of nanotubes is manifested by TEM images [35]. In Figure 3, TEM images of A (SWNT–COOH) and B (SWNT–(E)-4-(2-phenyldiazenyl)-3-hydroxynaphthalene-2-sulfonic acid) and C (SWNT-3–hydroxy –4 –nitrosonaphthalene–2–sulfonic acid) are shown. It indicates that the A has a smooth surface. The changes in the morphology for B and C are remarkable (Figure3). It seems that the diameters of B and C are slightly increased in comparison to A.

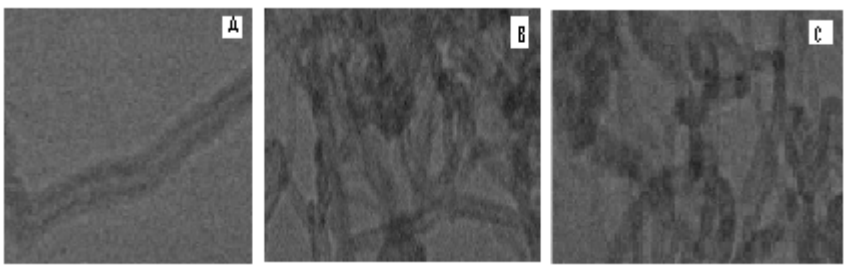

Figure 3 : TEM images of (A) and (B) and (C)

The functionalization of SWNT can be confirmed by the UV-visible spectra shown UV spectra of SWNT–COOH (A) and SWCNT-–(E)-4-(2-phenyldiazenyl)-3-hydrox ynaphthalene-2-sulfonic acid (B) and SWNT-3–hydroxy –4 – nitrosonaphthalene–2–sulfonic acid(C) were recorded as 1 mg in 1000 cc H_2O, λ_{max} and A (Absorbance) summarized in Table 1. The increase of λ_{max} in B and C were assigned to transmission electron of $\pi \longrightarrow \pi^*$in N=N and N=O in water soluble pigments (see the supporting information).

Name	λ_{max} (nm)	A
A	202	0.154
B	432	0.056
C	235	0.252

Table 1: λ_{max} and A of SWNT–COOH (A) and SWCNT–(E)-4-(2-phenyldiazenyl)-3-hydroxynaphthalene-2-sulfonic acid (B) and SWNT-3–hydroxy –4 – nitrosonaphthalene–2–sulfonic acid(C)

The chemistry of nanotubes offers considerable scope for development of functional materials, structures and devices based on SWNTs. A detailed methodology for the modification and functionalization of single walled carbon nanotube via esterification has been presented. We have introduced water soluble pigments on the surface of nanotubes. The functionalized SWNTs was demonstrated by SEM images, FT-IR, Raman spectroscopy and UV analysis, the results show successful functional groups.

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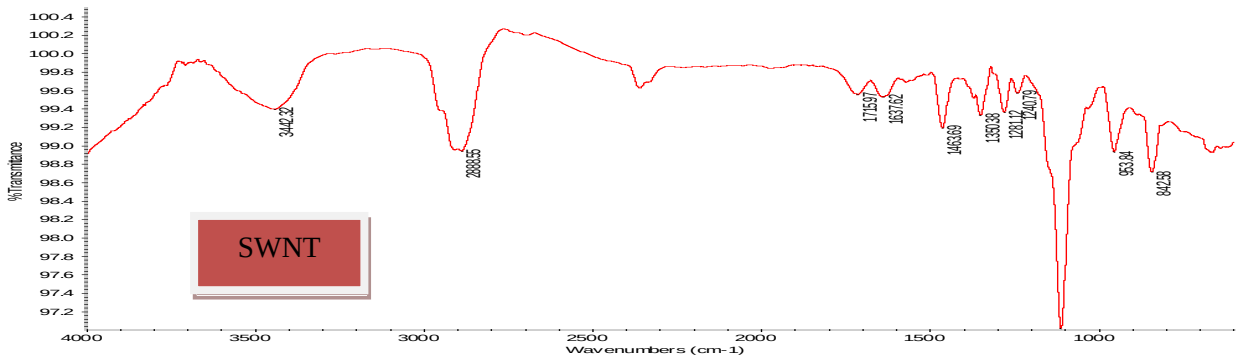
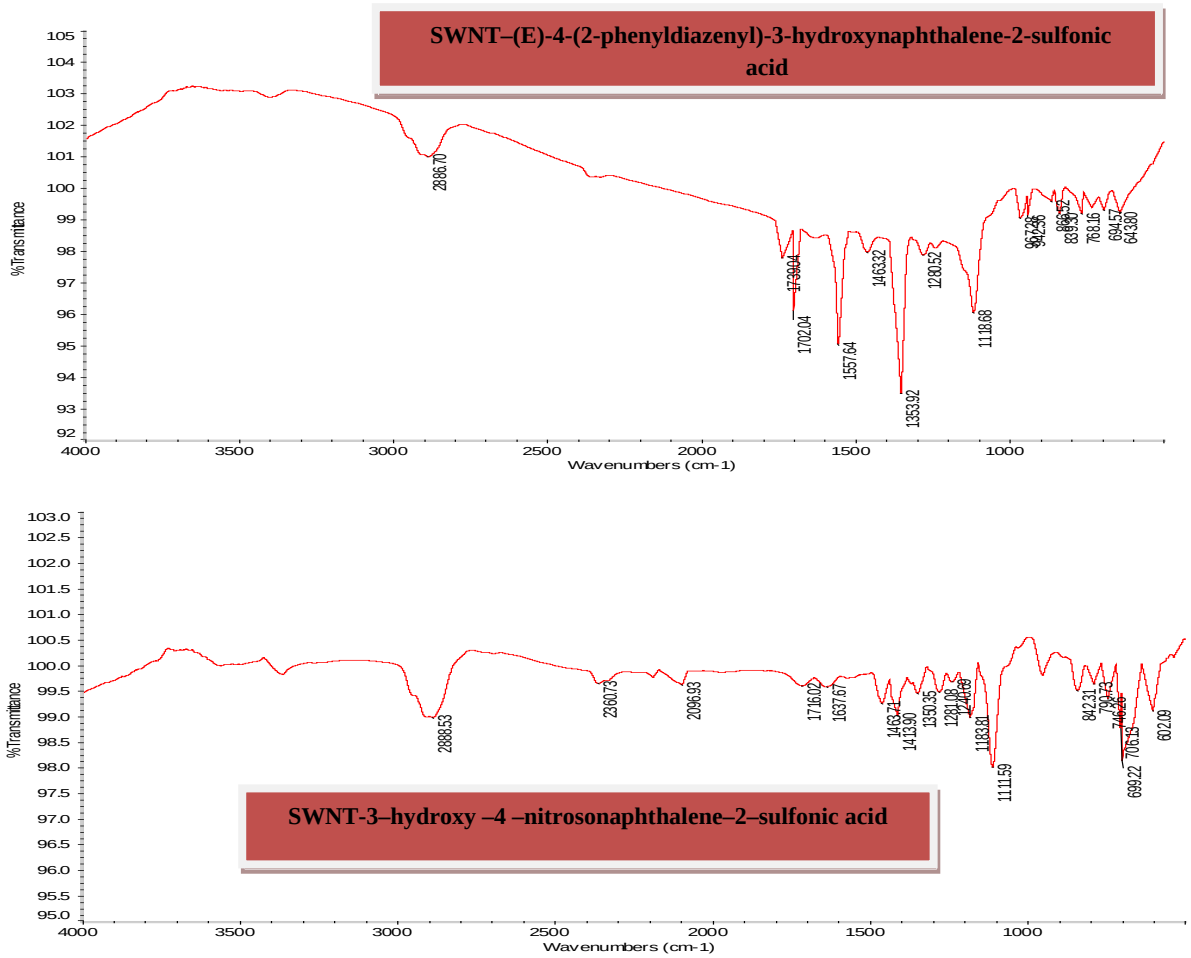
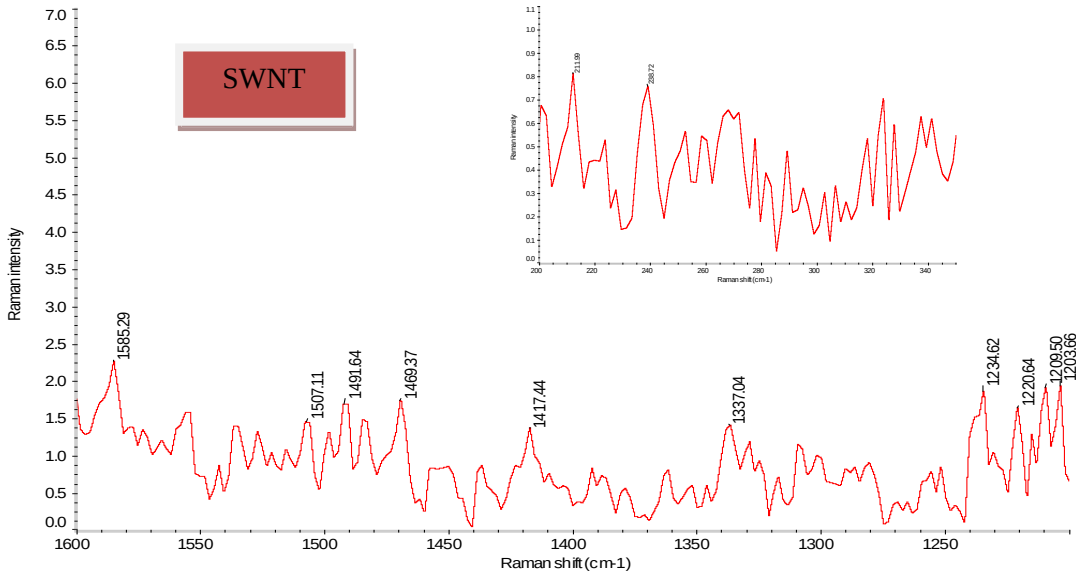
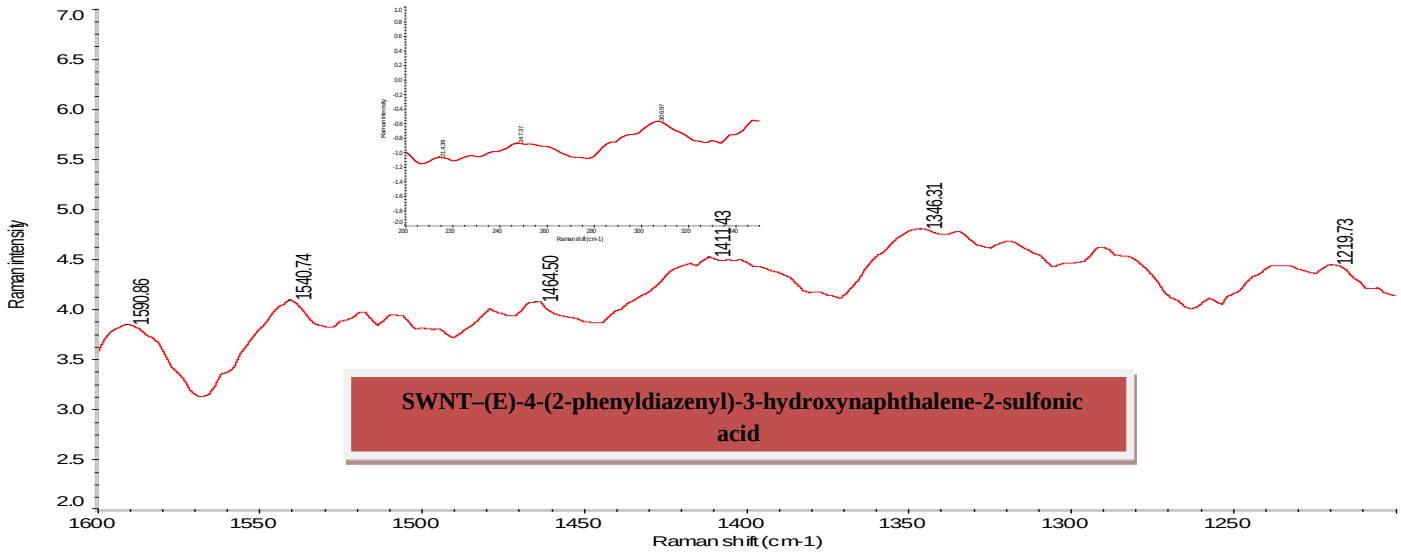




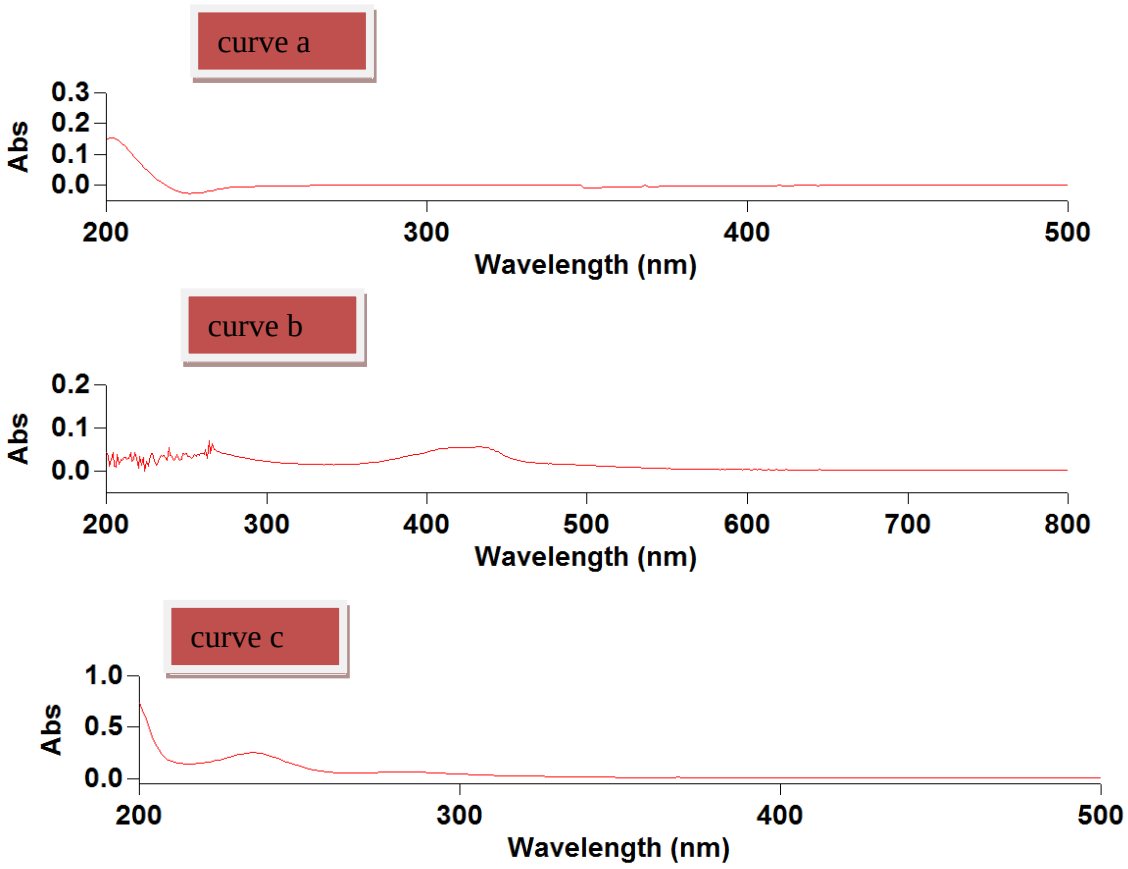
IR spectra for SWNT and SWNT–(E)-4-(2-phenyldiazenyl)-3-hydroxynaphthalene-2-sulfonic acid and SWNT-3–hydroxy –4 –nitrosonaphthalene–2–sulfonic acid





Raman spectra for SWNT and SWNT–(E)-4-(2-phenyldiazenyl)-3-hydroxynaphthalene-2-sulfonic acid and SWNT-3–

Ultra Violent images of (A) and (B) and (C)



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The One-Pot Three Components of Iodone, Schiff Base and Epoxide for Synthesis Iodohydrins

By Abolghasem Shameli, AbdolHamid Raeisi, Bahram Pourhasan, Hossein Naeimi & Mohmmad Mehdi Ghanbari

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Keywords : *Schiff base, Iodohydrin, ligand, iodine, homogeneous.*

GJSFR-B Classification : *FOR Code: 030299*

Strictly as per the compliance and regulations of :

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Abstract - In study we report synthesis Iodohydrines in absence of the complexes Schiff-base Salen **Cu, Ni** and **Co.** The experimental results showed both that the catalysts had higher catalytic activity and better epoxide selectivity than the homogeneous catalyst.

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I. Introduction

With the increasing application of rare earth metals in a variety of fields, rare earth ions continually intrude into general environment and further into the bodies of plants, animals and human beings. It is therefore of significance to investigate the physiological action and long-term effect of rare earth ion on biological bodies. Various studies have shown that Schiff bases derived from Salicylaldehyde and its derivatives have considerable biological importance partly because such ligands have many donor atoms (N, 0) and are analogous to biological environment to some extent. They have been widely used in the fields of biology, pharmacology, catalysis, organic synthesis, chemical, analysis, and so on [1-4]. much attentions have been paid to these Schiff bases because of the stability of the ligands and various properties of their metal complexes [5-10].

As an important strategy for the formation of 1,2-bifunctionalized chiral building blocks, the enantioselective ringopening of epoxides with different nucleophiles has attracted much attention from the organic chemists. A wide variety of nucleophiles, such as alcohols, phenols, carboxylic acids, amines,azide ions thiols, cyanide ions and halide ions are utilized in the aforementioned reaction[11].

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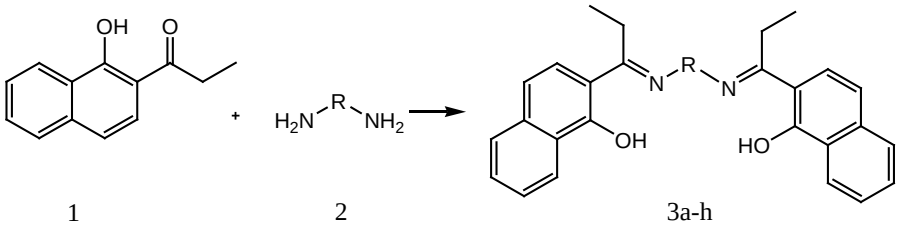
In continuation of our work on enantioselective epoxidation [12–17] of non-functionalized olefins by chiral Co(II), Ni(II) and Cu(II) Schiff base complexes and in pursuit of better selectivity through electronic tuning in the catalyst, we are reporting here the applying catalyst in ring opening opexides.

II. Experimental

All of the reagents were supplied by Merck and Fluka, and were employed without further purification. IR spectra were recorded on a Unicam Matson 1000 FT-IR paragon 1000 spectrophotometer. 1H NMR spectra of the ligand and the complex were recorded on a Bruker FT-NMR 500 MHz spectrometer using CDCl3 and (CD3)2SO as solvents. The electronic spectra were recorded on a CARY 100 Bio UV–Vis spectrophotometer.

a) General Procedure For Synthesis of 2,2'-[1,2-Ethandiyl Bis (nitrilopropelidene))]Blis(1-NAPHTHOL:

To the stirred solution of 1-(2-hydroxyznaphthal en-3-yl)propan-1-one (4 mmol) in 5ml MeOH Ethylendiamine (2 mmol) was added at room temperature. The reaction was continued for 3.4 h. The progress of the reaction was monitored by TLC. After the reaction was completed, the brown oil was collected and dissolved in hot petroleum ether. After cooling pail yellow solid product was obtained. The precipitate was filtered off and washed with cold MeOH. The crude product was purified by recrystallization in ethanol and the pure Schiff base, 2,2'-[1,2-Ethandiyl bis(nitrilopropelidene))]bis(1-naphthol) was obtained in 92% yield, m.p=196-198°C. The structure of the Schiff base was confirmed by physical and spectroscopic methods.



1 2 3a-h

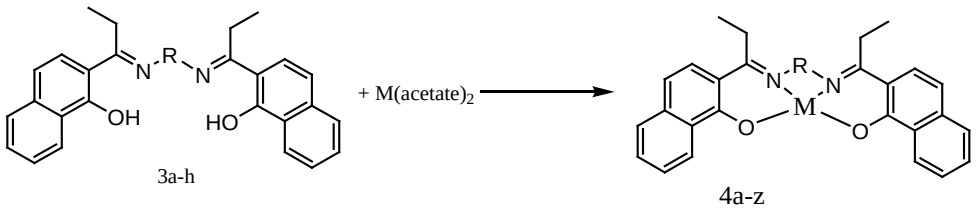
Scheme1: Synthesis Schiff base under condition room temperature

2,2'-[1,2 - Ethandiylbis (nitrilopropelidene)] bis (1-naphthol) (3a): Pail yellow, M.P=196-198 0C , IR(KBr)/ ν (cm-1): 3300-3550 (OH), 1600 (C=N), 1540 (C=C, Ar), 1H NMR/CDCl3 δ p.p.m; 1.2-1.6(6H, t, J=7.2), 3(4H, dd, J=7.2) ,4.2 (4H, s)7-8.4 (10H ,m), 8.6 (2H, d), 16.8(2H, s), λmax: 322 nm.

2,2'-[1,3-propandiyl is (nitrilopropelidene)] bisui (1-naphthol) (3b): Pail yellow, M.P=196-198 0C , IR(KBr)/ ν (cm-1): 3250-3550(OH), 160 (C=N), 1540 (C=C, Ar), 1H NMR/CDCl3 δ p.p.m; 1.2-1.6(10H, m), 3 (4H, m) ,4.2 (4H, s), 7-8.4 (10H ,m), 8.6 (2H, m), 16.8(2H, s), λmax: 330 nm.

b) Preparation of the Complexes

Ethanolic solutions of metal acetate (0.025mol) and schiff base (0.05mol) were mixed and the resulting mixture under conditions N2, until the metal Salen separated, which were then suction filtered, washed with ethanol and ether before dried in vacuum dessicator. The crystals were recrystallized from rectified sprit and dried (Mahaptra et al., 1977) (scheme1).



3a-h 4a-z

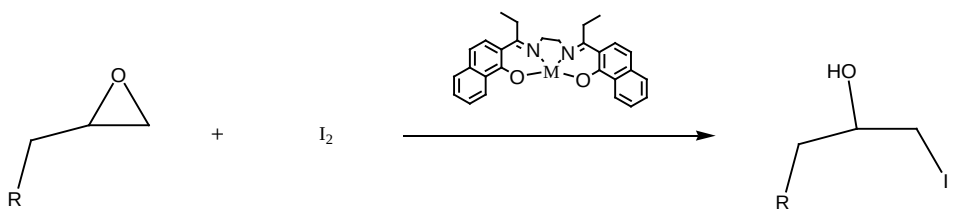
Scheme 2 : Synthesis Schiff base complex with Co, Cu and Ni

2,2'-[1,2-Ethandiylbis(nitrilopropelidene)]bis(1-naphthol ate) Copper(II) (4a): solid brown, M.P = 335 - 3370C ,IR (KBr)/ ν (cm-1): 1630 (C=N), 1550 (C=C, Ar), λmax: 318 nm

2,2'-[1,2-Ethandiylbis(nitrilopropelidene)]bis(1-naphthol ate) Cobalt(II) (4b): solid brown, M.P=337-33390C , IR (KBr)/ ν (cm-1): 1635 (C=N), 1560 (C=C, Ar), λmax: 320 nm

2.3.Synthesis bromohydrin by catalyst 2,2'-[1,2-Ethandiylbis (nitrilopropelidene)] bis (1 – naphtha – olate) Cobalt(II)

Epoxide (1 mmol) in CH2Cl2 (5 mL) was added to a stirred 2,2' - [1, 2 - Ethandiyl bis (nitrilo propelidene)]bis(1-naphtholate)Cobalt(II) catalyst (0.05 mmol) in at room temperature. Next, a solution of elemental iodine (1 mmol) in CH2Cl2 (5 mL) was added portion-wise (15 min) to the above mixture. The progress of the reaction was monitored by TLC. After complete disappearance of the starting material, the reaction mixture was washed with 10% aqueous Na2S2O3 (2×10 mL) and water (2×10 mL). The aqueous layer was extracted with CH2Cl2 (2×10 mL).The combined organic layer was dried over anhydrous MgSO4 and evaporated to give crude alcohol–catalyst (scheme3).



Scheme 3 : Synthesis iodohydrin with catalyst Schiff base

1-Iodo-2-boutanol (99%): 1HNMR (CDCl3, 300MHz) δ 0.95 (t, 3H, J=7.2Hz), 1.55-1.7 (m, 2H), 3.05-3.25(m, 1H), 3.3-3.5 (m, 1H), 3.75-3.8(m, 1HNMR (CDCl3, 300MHz) δ 2.50(br, 1H), 3.39-3.5 (m, 2H), 4.75(m, 1H), 7.25-7.4(m, 5H);MS(EI) M/Z 248(M+); IR(KBr) 3398 , 2960 cm-1

1-Iodo-3-(4-methoxyphenyl)2-propanol (99%): pale yellow liquid, 1HNMR (CDCl3, 300MHz) δ 2.05(br, 1H), 2.85(d, 2H, J=6.2, 9.2Hz), 3.25 (dd, 1H, J=4.8,9.2 Hz), 3.35 (dd, 1H, J=3.8,9.2Hz), 3.6-3.75(m, 1H), 3.80(S, 3H),6.85(d, 2H,J=8.2Hz), 7.15(d, 2H, J=8.2Hz); 13CNMR (CDCl3,50Hz) δ 14.67, 41.66, 55.10, 71.62,

113.92, 128.98, 130.12, 158.25;MS (EI) M/Z 292 (M+); IR(KBr) 3560, 3050, 2960 cm^{-1}

1-Iodo-3-(4-acetylphenoxy)2-propanol (99%): yellow Solid, mp 68-700 C, 1HNMR (CDCl3, 300MHz) δ 2. 55(S, 3H), 3.30-350 (m,2H), 3.90-4.05 (m, 3H), 6.90(d, 2H,J=7.8Hz), 7.9(d, 2H,J=7.8Hz) ; 13CNMR (CDCl3,50Hz) δ 8.84, 26.21, 69.12, 70.15, 114.66, 130.64, 162.75, 196.96; MS (EI) M/Z 320 (M+); IR(KBr) 3460, 3020, 2970, 1710 cm-1

1-Iodo-3-(4-Cholorophenoxy)2-propanol (98%) : 1HNMR (CDCl3, 300MHz) δ 2.40 (br, 1H), 3.35-3.40 (m,2H), 3.45-3.50 (m, 1H), 3.95-4.0 (m, 1H), 4.05-4.10(m, 2H), 6.85(d, 2H, J=8.2 Hz), 7.15(d, 2H, J=8.2 Hz); MS (EI) M/Z 312 (M+); IR(KBr) 3515 cm-1

1-Iodo-3-phenoxy-2-propanol(97%): 1HNMR (CDCl3, 300MHz) δ 2.40(br, 1H), 3.30-3.55 (m, 2H), 3.8-4.1(m, 3H), 6.75-7.0(m, 3H) 7.15-7.35(m,2H);MS(EI) M/Z 278(M+); IR (KBr) 3500 , 2985 cm-1 .

(m,2H), 3.45-3.50 (m, 1H), 3.95-4.0 (m, 1H), 4.05-4.10(m, 2H), 6.85(d, 2H, J=8.2 Hz), 7.15(d, 2H, J=8.2 Hz); MS (EI) M/Z 312 (M+); IR(KBr) 3515 cm-1

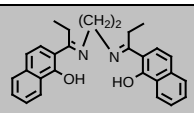
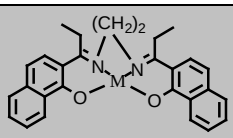
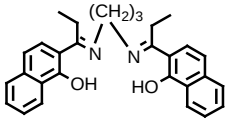
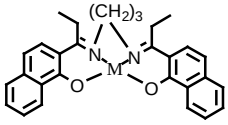
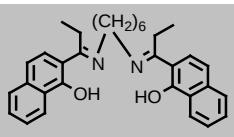
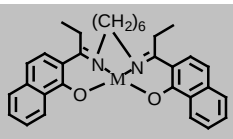
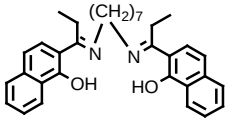
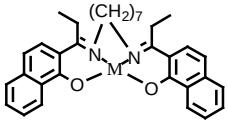
1-Iodo-3-phenoxy-2-propanol(97%): 1HNMR (CDCl3, 300MHz) δ 2.40(br, 1H), 3.30-3.55 (m, 2H), 3.8-4.1(m, 3H), 6.75-7.0(m, 3H) 7.15-7.35(m,2H);MS(EI) M/Z 278(M+); IR (KBr) 3500 , 2985 cm-1 .

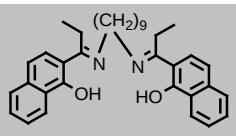
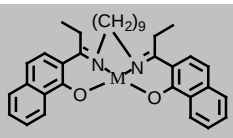
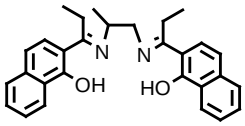
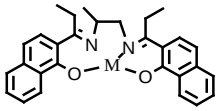
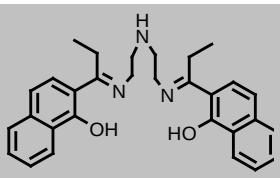
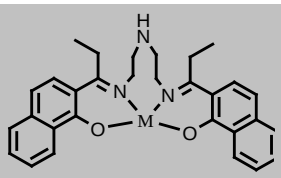
III. Result and Discussion

As shown in Scheme 1, when 2 mols heterocyclic ketone were treated with 1 mol diamine at room temperature, a pail yellow substance obtained with high yield. In this reaction, heterocyclic ketones have been applied and corresponding products were obtained. The results and conditions of the reactions are presented in Table 1.

As can be seen in our previously reported works on synthesis of Schiff bases from ortho-hydroxy aldehyde [14-16] and ortho-hydroxyl ketene, [16-17] the presence of hydroxyl group in ortho situation is accelerated condensation reaction. While, in used heterocyclic aldehydes, with respect to absence of o-hydroxy group, the corresponding Schiff bases were obtained in high yields and appropriate reaction times (Table 1, Entries 1, 2).

Table 1 : Preparation of Schiff base containing heterocyclic rings through two component reaction

Entry	Schiff base	complex	Time(min) [(a= Schiff base) complex(b= Cu c= Co d=Ni)]	Yielda (%)
1			a=10 b=20 c=20 d=20	a=93 b=95 c=95 d=95
2			a=20 b=30 c=30 d=30	a=95 b=90 c=90 d=85
3			a=20 b=35 c=35 d=35	a=90 b=80 c=87 d=85
4			a=20 b=35 c=35	a=85 b=87 c=88

			d=35	d=88
5			a=20 b=40 c=40 d=40	a=85 b=75 c=75 d=75
6			a=30 b=20 c=20 d=20	a=85 b=80 c=87 d=85
7			a=30 b=60 c=60 d=60	a=85 b=80 c=75 d=78

alsolated product yields

All the new, potentially hexadentate Schiff base ligands were cleanly synthesized in 1-1.5 minutes and >80% yield according to elemental analyses and 1H and 13C NMR analyses of the bulk products after recrystallization from ethanol (Table 1). Their structures are supported by the absence from their IR spectra of the carbonyl and primary amine bands of the reagents, and the presence of a Schiff base ν(C=N) band in the 1631-1652 cm-1 region; the alkyl C–H stretching vibrations appear in the 2800–2900 cm−1 region. In the 1H NMR spectra, the azomethine protons appear at δ= 8.22–8.73 ppm and the aromatic ring protons at δ = 6.5-8.4 ppm.

In conjunction with ongoing work in our laboratory on the synthesis and formation of complex heterocyclic compounds containing donor nitrogen atoms, with neutral molecules such as bromine, we found out that 2,2'-[1,2-Ethandiylbis (nitrilopro peli dene)]bis(1-naphth-olate) Cobalt(II) with frame nano efficiently catalyzed the addition of elemental bromine to epoxides under mild reaction conditions with high regioselectivity (Scheme 3).

In this study, we wish to report the results of the reactions of some styrene oxide with elemental bromine in the presence of a sub-stoichiometric 0.01 mmol amount of catalyst Schiff base (Scheme 3, Table 2).

Table 2 : Amount of catalyst in ring opening epoxide

Entry	Complex Schiff base	Yielda (%)	Entry	Complex Schiff base	Yielda (%)	Entry	Complex Schiff base	Yielda (%)
1	4a	40	8	4h	15	15	4q	10
2	4b	70	9	4k	10	16	4r	5
3	4c	25	10	4l	10	17	4s	5
4	4d	30	11	4m	20	18	4t	10
5	4e	15	12	4n	10	19	4w	5
6	4f	20	13	4o	10	20	4x	5
7	4g	10	14	4p	10	21	4y	5

alsolated product yields

Then in this article, we report ring opening styrene oxide with amount deferent of catalyst 4b, that the results of reaction showing in table 4.

Table 3 : Cleavage styrene oxide with amount deferent of catalyst 4b

Entry	Complex 4b (% mol)	Yielda (%)	Entry	Complex 4b (% mol)	Yielda (%)	Entry	Complex 4b (mol)	Yielda (%)
1	1	70	4	4	90	7	7	100
2	2	75	5	5	98	8	10	100
3	3	80	6	6	98	9	15	100

alsolated product yields

The crude products were purified on a column of silica gel. The solvent was evaporated and pure iodohydrin was obtained. The iodohydrins obtained throughout this procedure were identified by comparison, where possible, with authentic samples prepared in accordance with literature procedures.

IV. Conclusion

In conclusion, this new method appears to be highly competitive with the other methods reported in the literature. The reaction occurs in neutral and mild conditions on the acid-sensitive substrates and vicinal iodohydrins were obtained in high yields and regioselectivity. In addition, in comparison with our previous methods, 2,2'-[1,2-Ethandiylbis (nitrilo pro peli-dene)] bis(1-naphtholate) Cobalt(II) is cheaper, less step need for preparation, and overall yield is higher

Acknowledgment

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	A-B	**C-D**	**E-F**
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Introduction	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
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Result	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
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References	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring

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