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GLOBAL JOURNAL

OF SCIENCE FRONTIER RESEARCH: B

Chemistry

Highlights

- Physico-Chemical Analysis
- Physical Properties of Polymer
- Domino Reaction of Indoles
- PCP Removal by Sulfate Radical

Discovering Thoughts, Inventing Future

VOLUME 14 ISSUE 6 VERSION 1.0

Global Journal of Science Frontier Research: B

Chemistry

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Physico-Chemical Analysis for the Presence of Oxygen Content of Ground Water at Different Locations of Dildar Nagar of U.P, India

By Salahuddin & R. K. Khola

Mewar University, India

Abstract- In this paper, we discuss about the recently collected sample of ground water at different locations in Uttar Pradesh State of India and its experimental analysis in laboratory for the presence of Oxygen content. Also, we represents the data graphically and interpreted the data using the method called analysis of variance. Further, we analyze our findings with the established results. Lastly we concluded that the samples depend on location as well as month also that is in different months and different areas disolved Oxygen are different.

Keywords: ground water; analysis of variance; graphical representation.

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I. INTRODUCTION

Groundwater is the water located beneath the earth's surface in soil pore spaces and in the fractures of rock formations. A unit of rock or an unconsolidated deposit is called an aquifer when it can yield a usable quantity of water. The depth at which soil pore spaces or fractures and voids in rock become completely saturated with water is called the water table. Groundwater is recharged from, and eventually flows to, the surface naturally; natural discharge often occurs at springs and seeps, and can form oases or wetlands. Groundwater is also often withdrawn for agricultural, municipal, and industrial use by constructing and operating extraction wells. The study of the distribution and movement of groundwater is hydrogeology, also called groundwater hydrology.

a) Dissolved Oxygen

The DO is Required by higher forms of aquatic life for survival. The DO standards were formulated considering fisheries criteria. The national comnuittee on water pollution control resources established guidelines in 1958 for water use DO. Relatively good water bodies have more than 7.5 mg/l. For fisheries, hatching of salmon and trout rearing, more than 7 mg/l DO is required. Other general aquatic organisms also require more than 6 mg/l. In Ohio State, USA, the DO standard for fisheries is 5 mg/l. The Japanese standard for class 3 fisheries is established at the same level. Dissolved oxygen should be more than 5 mg/l for agriculture use, because DO less than 5 mg/l interferes with root growth. The DO level for the conservation of the environment should be kept at more than 2 mg/l to prevent anaerobic conditions that cause bad odors.

b) Anova

It is a statistical tool used in several ways to develop and confirm an explanation for the observed data. It is an extension of the t-test, which is used in determining the nonsignificance of difference of three or more group of values.

The calculations of ANOVA can be characterized as computing a number of means and variances, dividing two variances and comparing the ratio to a handbook value to determine statistical significance.

The F-test is used for comparisons of the components of the total deviation. For example, in one-way or single factor ANOVA, statistical significance is tested for by comparing the F test statistic
F=Variance between samples/ Variance within samples.

The textbook method of concluding the hypothesis test is to compare the observed value of F with the critical value of F determined from tables. The critical value of F is a function of the numerator degrees of freedom, the denominator degrees of freedom and the significance level (α). If F FCritical (Numerator DF, Denominator DF,α) then reject the null hypothesis.

c) Sample Collection

The samples of ground water are collected from various areas near to Dildar Nagar in Uttar Pradesh state. Samples are collected in plastic container to avoid unpredictable changes.

d) Physico-Chemical Analysis Of Ground Water

The collected samples were analyzed for disolved Oxygen which are collected from different areas and different months near to Dildar Nagar.

II. RESULTS AND DISCUSSION

The water quality analysis of different locations of ground water samples have been carried out for disolved oxygen. The status of water quality of ground water sources for dissolved Oxygen are presented in Table 1 and represented graphically in Fig 1.

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The disolved Oxygen in various areas according to various months are different which is estabished by statistical analysis.

Table 1 : Monthly variation in Dissolved Oxygen (mg/l) of ground water at different sampling sites

CODE	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEPT	OCT	NOV	DEC
GW1	5.1	5.2	6.5	6.2	5.7	5.5	5	4.9	4.5	5.6	5.7	5.3
GW2	5.3	5.5	5.6	5.4	5.6	5.1	4.9	4.3	4.2	5.1	5.2	5.2
GW3	5.4	4.8	4.7	4.5	5.3	4.4	3.8	3.8	3.3	3.1	3.2	3.9
GW4	5.9	6	6.1	6.3	6.1	5.6	5.2	5.1	5.2	5.6	5.6	5.7
GW5	5.1	5.2	5.1	5.4	5.7	5.7	5.4	4.8	4.9	5.1	5.2	5.2
GW6	5.7	5.2	5.1	5.4	5.3	5.4	4.9	4.7	4.1	4.2	4.9	4.8
GW7	5.2	5.1	5	5.6	5.7	5.6	5	5.2	5.5	5.6	5.4	5.6
GW8	5.1	5.2	5.7	5.7	5.8	5.7	6.3	6.2	5.6	6.1	6.1	6.4
GW9	5.1	5.4	5.7	6.2	6.1	6.1	5.6	5.9	6.2	5.5	6.1	5.8
GW10	6.2	6.1	6.7	6.1	6.2	6.2	5.2	5.4	5.4	5.8	6	5.2

GW1= Yuduf Pur, GW2 = Dildar Nagar, GW3 = Saidpur, GW4 = Nandganj Railway Station, GW5 = Attarsua Village, GW6 = Reonsa village, GW7 = Dhamupur, GW8 = Saheri village,GW9 = Kusmhi Kala village, GW10 = Husainpur village

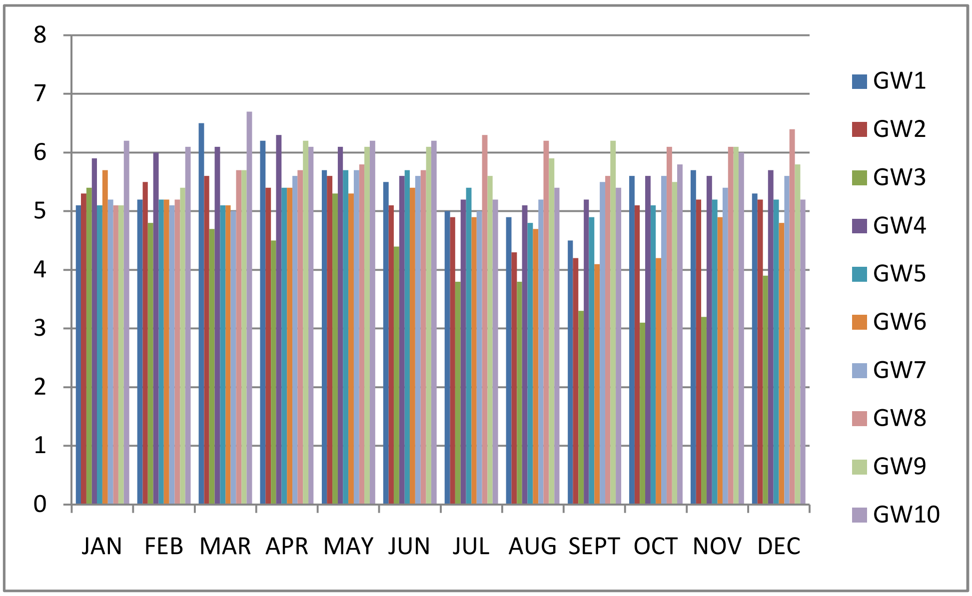

Fig. 1 : Graphical representation of the data

a) Analysis the Data using two way Anova

Sum of squares between areas= 7.74625

Sum of squares between months = 28.93342

Sources of value	Sum of squares	Degrees of freedom	Mean square(variance)
Between Area	7.74625	11	0.704205
Between month	28.93342	9	3.214824
Residual	16.35958	99	0.165248
Total	53.03925	119	

Let us take the Hypothesis that there is no signaficance difference of Oxygen content between the areas and months.

First we compare the variance of areas with the variance of residual.

$F_1 = 4.261493$.

The table value of F_1 for $\upsilon_1=11$ and $\upsilon_2=99$ at 5% level of significance is 1.886684.

The calculated value is greater than table value and we conclude that the Oxygen content of different areas are not same that is Oxygen content of different areas are different.

Now , let us compare the variance according to months with the variance of residuals.

$F_2= 19.4545$.

The table value of F_2 for $\upsilon_3=9$ and $\upsilon_2=99$ at 5% level of significance is 1.975806.

The calculated value is greater than table value and we conclude that Oxygen content of different areas changes according to months. That is Oxygen content depends on months.That is Oxygen content in different month is different.

III. CONCLUSION

It is concluded that the Oxygen content of ground water depends on locations as well as months. In Saidpur it is not suitable for agriculture use except the month of January because DO less than 5 mg/l interferes with root growth.

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Storage and Operational Stability of pH-Responsive Hydrogels

By Jeffrey Bates & Jules Magda
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Introduction- Hydrogels have proven their ability to respond to changes in the local environment [1-6]. While the results obtained by many researchers highlight the promising nature of hydrogels in biomedical sensors, work has yet to be done to demonstrate the ability of hydrogels to maintain a response after being stored for an extended period of time, and to demonstrate the ability to maintain a strong stimuli response after repeated cycles. Some researchers have proposed utilizing hydrogel-based sensors in implantable devices [7]. If this technology is to work, it is important to understand the duration and stability of the stimuli response. This will determine the life of a hydrogel-based sensor and the time frame in which the device will become ineffective and need to be replaced. Furthermore, devices may not be used as soon as the hydrogel has been synthesized. Therefore, it is also important to understand how long a device may remain in storage before it loses its effectiveness.

GJSFR-B Classification : FOR Code: 039999.

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A hydrogel with a 2-hydroxyethyl methacrylate (HEMA) backbone was studied to determine the ability of the hydrogel to respond after extended periods of time in ambient conditions. The time intervals for this study were at 0, 9, and at 18 months. The data gathered in this study will be useful in determining storage duration and conditions for maintaining a strong stimuli response of the hydrogel.

This study also addresses the operational stability of the hydrogel. This will help researchers determine the length in which a hydrogel-based chemomechanical sensor may be used in medical and other biological applications without losing sensitivity to changes in environmental conditions.

Here data are presented that have been gathered at set time intervals (0 months, 9 months, and 18 months after hydrogel synthesis) and with prolonged testing (up to 300 cycles). The tests performed on the HEMA hydrogel were under ionic strength conditions. HEMA hydrogels are known to respond to multiple analytes, including pH and ionic strength [8-11]. The ionic strength response is fast (3-5 minutes), and shows a clear stimuli response to small changes in ionic strength concentrations. Furthermore, the ionic strength response is used here for rapid cyclic testing.

II. Experimental Methods

a) *Materials*

The following monomers were used as received from Sigma Aldrich: 2-hydroxyethyl methacrylate (HEMA), dimethylaminoethyl methacrylate (DMA), and tetraethylene glycol dimethacrylate. In addition, 2,2-dimethoxy-2-phenylacetophenone (DMPAP), a photoinitiator, and ethylene glycol (EG), a solvent for the pregel solution, were also obtained from Sigma Aldrich and used as received. Dulbecco's phosphate buffered saline was also obtained from Sigma Aldrich and mixed at 9.6 g/L in deionized water.

After preparation, hydrogel samples were tested with a piezoresistive sensor. A conductivity meter was used to measure the conductivity of the testing solution. An automated, continuous flow system comprising of a data acquisition device, pumps, and lab view software was used to change the concentration of the testing solution.

b) *Hydrogel Synthesis*

Hydrogel monoliths were synthesized in a mole ratio of 91.2 DMA, 1.1 HEMA, 0.2 TEGDMA, and 7.5 EG and a thickness of 400 μm. The hydrogel was conditioned by alternating concentrations of PBS every 4 hours for 3 cycles. The PBS concentrations were alternated between 55 mM and 165 mM PBS.

c) *Testing Conditions*

This hydrogel has been proven to swell in response to changes in ionic strength. The two testing conditions for the ionic strength test were from 155 mM PBS to 165 mM PBS. To obtain these concentrations, 9.6 g of PBS in powder form was added to 1 L deionized water and diluted to 155 mM and 165 mM concentrations.

d) *Testing Procedures*

The swelling pressure of the hydrogel samples was measured using a pressure sensor [12-14]. The pressure consisted of a piezoresistive sensor and a cap containing a porous mesh membrane. The cylindrical hydrogel sample (3.5 mm diameter and 400 μm height) was placed in the pressure sensor and placed into the testing conditions, starting at 155 mM. The continuous

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flow equipment was programmed to alternate the concentrations of PBS between 155 mM and 165 mM every 15 to 30 minutes.

e) Storage Stability

A shelf life test was designed to determine how long a hydrogel sensor could sit on a shelf in a clinic before it would no longer work. For this test, a hydrogel monolith was synthesized, and samples were tested at these time intervals: 1 week after synthesis, 9 months, and 18 months. Hydrogel samples tested at each time interval were performed under ionic strength conditions where the ionic strength of the media solution was changed between 155 mM and 165 mM. Experiments were performed for at least 3 cycles and the average values for the response time and magnitude were collected and analyzed.

f) Transportation and Signal Stability

Simulated transportation tests were performed on the hydrogels to determine the signal stability after transportation. Two hydrogel monoliths were prepared.

The first monolith, the control sample, was synthesized and immediately hydrated, washed, and conditioned as specified above. The second hydrogel monolith was synthesized and immediately placed in a 100 mL container. The container with the hydrogel sample was packaged in a padded mailing envelope and placed in a vehicle for 7 days and driven under normal conditions to simulate travel conditions. The hydrogel was then hydrated, washed, and conditioned as described above.

Both hydrogel samples were tested with the same conditions: 25 °C and 155 mM ionic strength. Solutions for this test were prepared by mixing 100 mL PBS solution with 0.1 M HCl to obtain pH levels of 7.2 and 7.4. The solutions were mixed by adding 500 μL of 0.1 M HCl under constant stirring with a calibrated pH electrode until the necessary pH readings were obtained for each solution.

The hydrogel samples from each monolith were tested with the same sensor for three cycles to determine the change in the response after simulated travel conditions.

g) Operational Stability

The second test was performed to determine if the stimuli response would decrease after multiple cycles of testing. The hydrogel samples were loaded and tested continuously in the pressure sensor with the automated flow system for up to 100 cycles under the same conditions listed above. The hydrogel samples in each experiment were stored in 165 mM PBS solution at room temperature for up to 18 months.

III. RESULTS

a) Storage Stability

After performing the same test on hydrogel samples taken from the same hydrogel monolith, the data were analyzed to determine the first order response time and the magnitude of swelling.

The following graphs illustrate one swelling and one deswelling cycle at each of the specified time intervals. The arrows on the graphs (see Figures 1.1-1.3) represent changes in the ionic strength concentration during the experiments. The hydrogel samples swell at low ionic strength concentrations and swell as the ionic strength concentration increases.

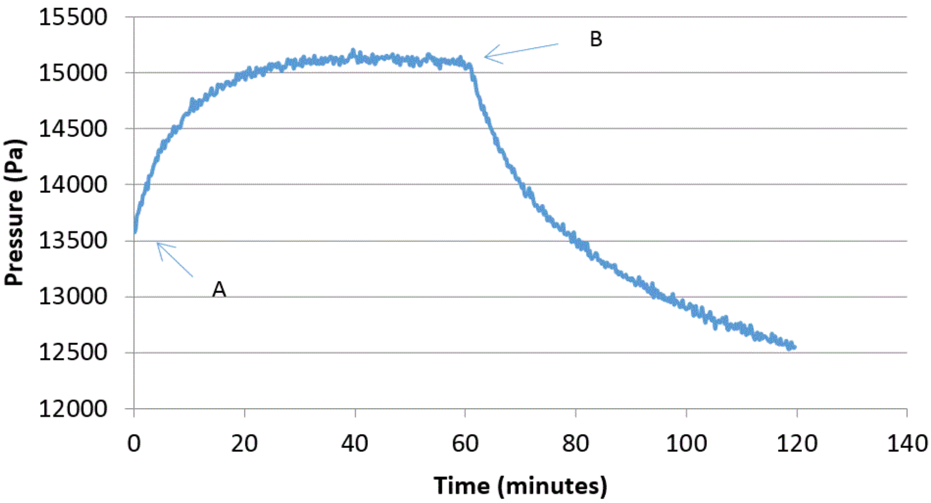

Figure 1.1 : An ionic strength test where the ionic strength concentration was decreased (from 165 mM to 155 mM) at point A. When the hydrogel sample came to equilibrium, the concentration was changed to a higher concentration at point B (from 155 mM to 165 mM). The average first order response time was 22 minutes for swelling and 17 minutes for deswelling. The average magnitude response change was 1.6 KPa for swelling and 2.8 for deswelling

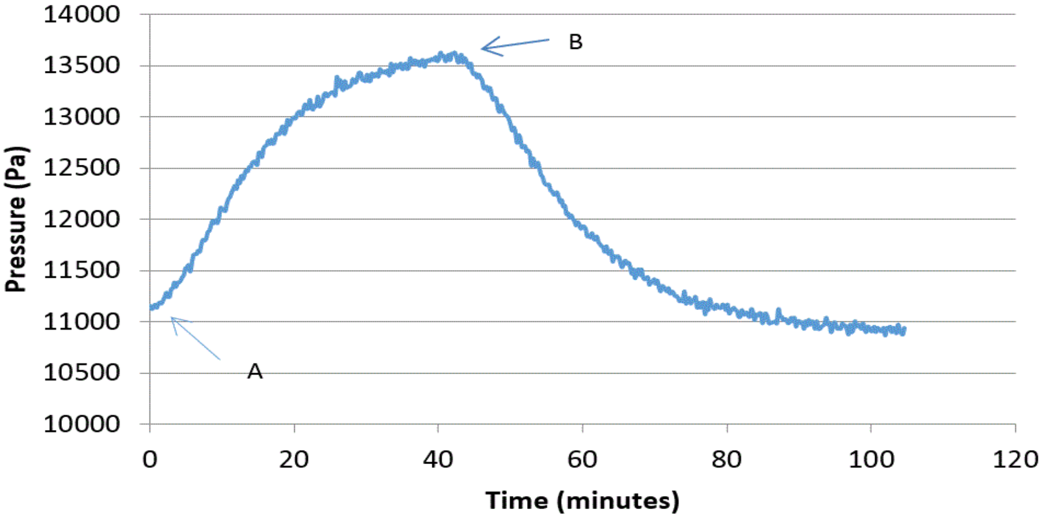

Figure 1.2 : An ionic strength test after 9 months where the average first order response time was 9 minutes for swelling and 14 minutes for deswelling. The average magnitude response change was 2.4 KPa for swelling and 2.6 KPa for deswelling

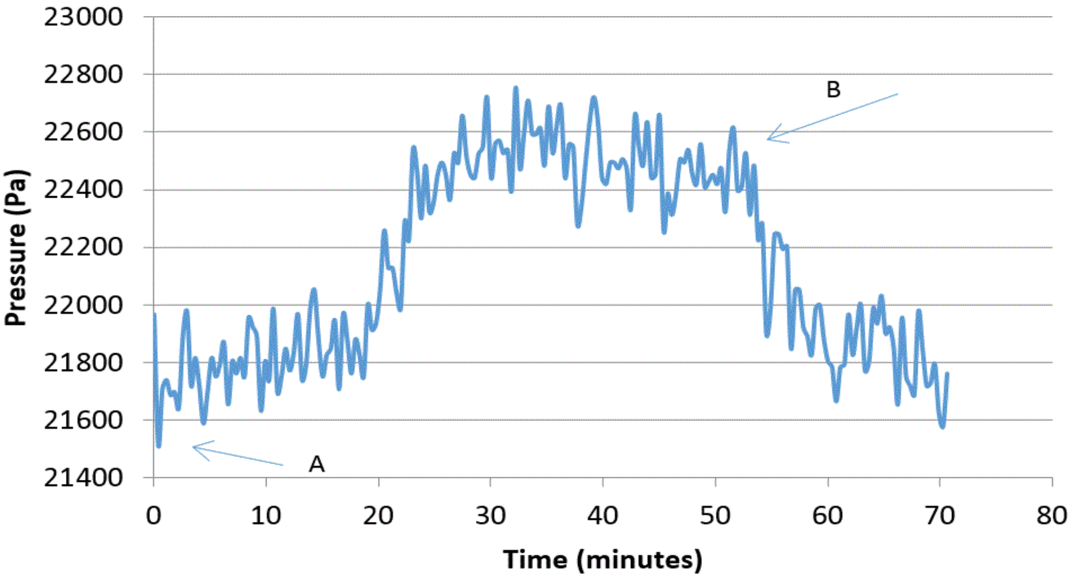

Figure 1.3 : An ionic strength test after 18 months where the average first order response time was 9 minutes for swelling and 8 minutes for deswelling. The average magnitude response change was 1.1 KPa for swelling and 1.6 KPa for deswelling

The data represented in these graphs illustrate that the hydrogel has the ability to respond to changes in ionic strength. Furthermore, they illustrate that the hydrogel continues to be responsive after being stored in a stock solution of PBS for extended periods of time.

b) Transportation Testing

The control hydrogel sample was tested under the conditions outlined above. The average response time for swelling was 74 hours with a magnitude of 6.2 KPa. The average deswelling response time was 45 hours with a magnitude of 5.6 KPa (see Figure 1.4).

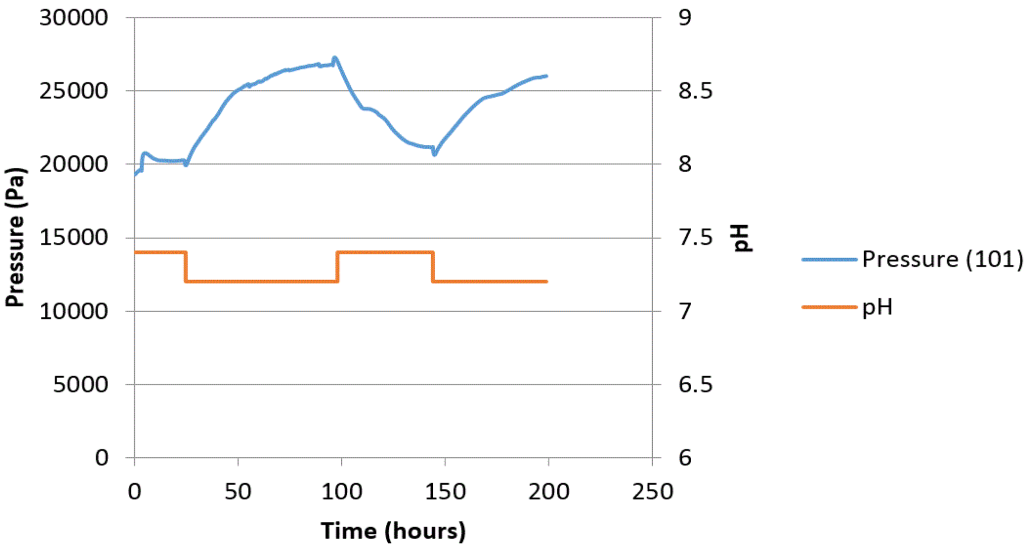

Figure 1.4 : The control test of the chemomechanical sensor showing the pH response from 7.2 to 7.4 prior to simulated transportation

The same test was performed on another hydrogel sample of identical composition in the same chemomechanical sensor after simulated transportation (see Figure 1.5). The average response time for swelling in this experiment was 35 hours with a magnitude of 19 KPa. The average deswelling response time was 28 hours with a magnitude of 17 KPa.

c) *Operational Stability*

The initial test was conducted within 1 week of synthesizing the hydrogel. The primary objective of this test was to determine the sensitivity of this hydrogel to small changes in ionic strength concentrations. The data show that the swelling magnitude is smaller than the deswelling magnitude (see Figure 1.6).

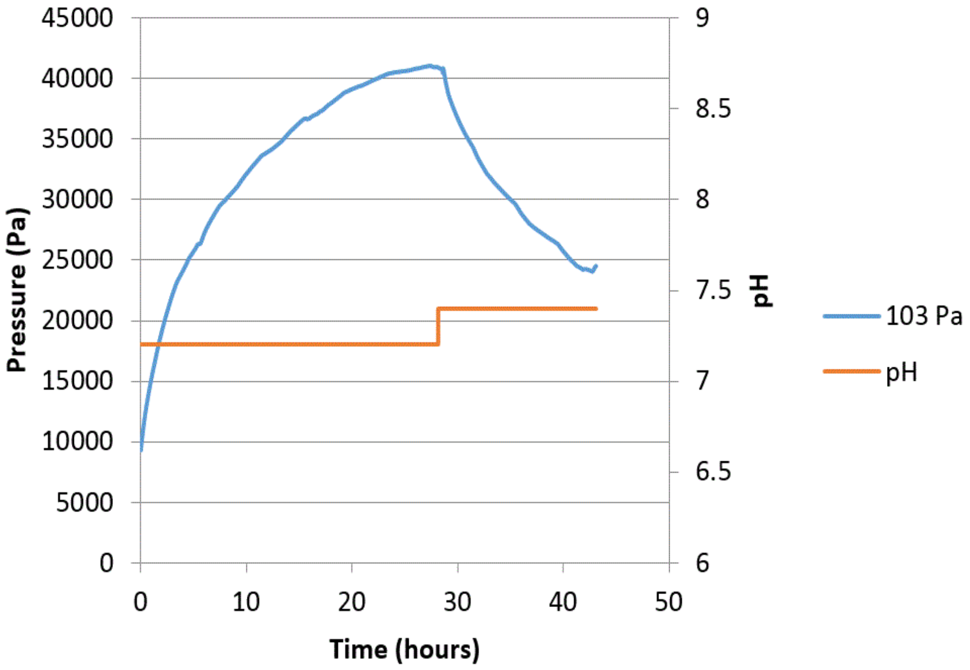

Figure 1.5 : A pH response test from pH 7.2 to 7.4 of the hydrogel sample in the M-Biotech sensor after simulating travel conditions

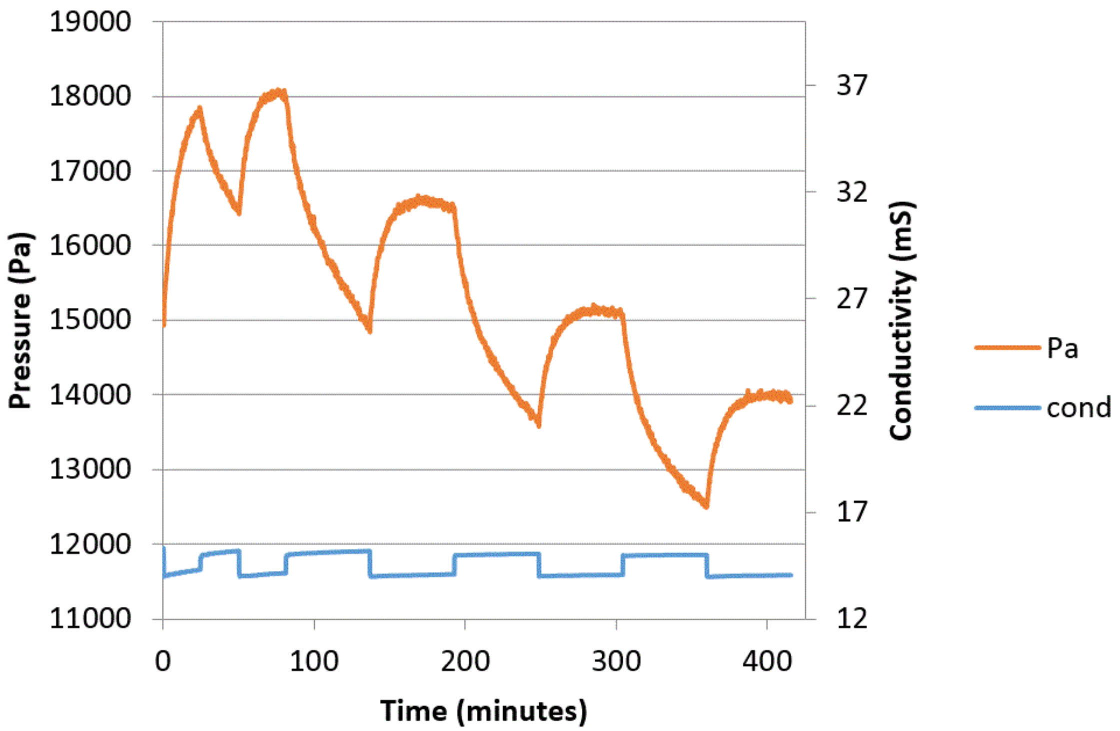

Figure 1.6 : An ionic strength test immediately after synthesis where the initial test was conducted within 1 week of the hydrogel synthesis. The hydrogel was tested for 5 continuous cycles. After the first 2 cycles, the continuous system was modified in order to allow the swelling and deswelling to come to an equilibrium point

The second test was performed after 9 months of storage. The objective of this test was to determine if the magnitude of the response to the change in ionic strength concentration would change after repeated testing (see Figure 1.7). The response magnitude and time were taken at different time intervals to determine the change (see Table 1.1).

Based on these data, the magnitude of the deswelling response was greater than that of the swelling response for the first 7000 minutes (40 cycles). In addition, the deswelling response time decreases as the number of cycles increases. As the swelling and deswelling response approaches equilibrium, there was no significant difference between the response times and magnitudes in either swelling or deswelling. However, there remained a difference between the swelling and deswelling response times, which was also noted in the test after one week of synthesis. After 40 cycles, the response of the hydrogel reached equilibrium, where the magnitude of the response for swelling was equal to the magnitude of the response for deswelling.

A sample of the hydrogel was tested again at 18 months to determine the response times and magnitudes at different time intervals. The objective of this test was also to determine if the response time and magnitude would change with multiple cycles. As the test at 9 months yielded no significant change as it approached equilibrium, it was decided to test this hydrogel with 100 cycles (see Figure 1.8). As with the test at 9 months, the response time and magnitude data were collected at set time intervals (see Table 1.2). This test showed that the hydrogel was tested through 25 cycles before the hydrogel was able to reach equilibrium. After the initial 25 cycles (1600 minutes) the hydrogel obtained stability, and the response times and magnitudes remained constant.

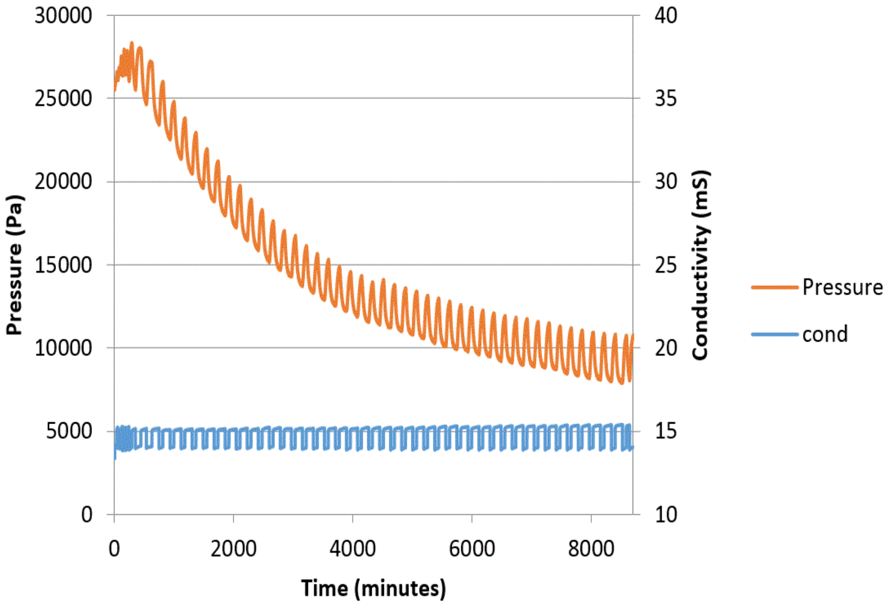

Figure 1.7 : An ionic strength test after 9 months where the hydrogel was tested for 51 continuous cycles

Table 1.1 : The swelling and shrinking response times and magnitudes at various time intervals illustrate the stable response of the hydrogel

Swelling			Shrinking		
Time (minutes)	Response Time (minutes)	Magnitude (KPa)	Time (minutes)	Response Time (minutes)	Magnitude (KPa)
1000	9	2.37	1000	19	3.35
3000	11	2.43	3000	19	2.77
5000	9	2.4	5000	14	2.63
7000	9	2.7	7000	15	2.85
9000	9	2.55	9000	13	2.75

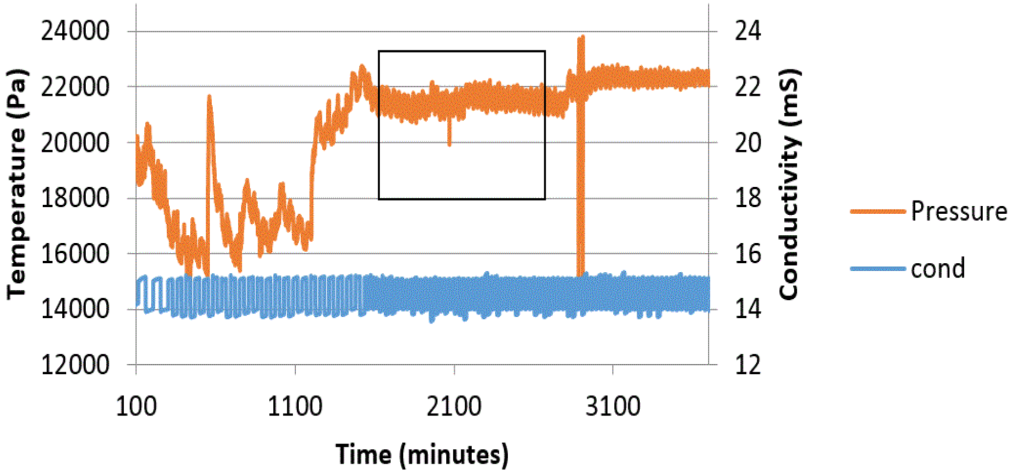

Figure 1.8 : An ionic strength test after 18 months of synthesis where the hydrogel was tested for 100 continuous cycles. The portion of the graph within the box is magnified in Figure 5.9.

Table 1.2 : The swelling and shrinking response times and magnitudes at various time intervals illustrate the stable response of the hydrogel

Swelling			Shrinking		
Time (minutes)	Response Time (minutes)	Magnitude (Kpa)	Time (minutes)	Response Time (minutes)	Magnitude (Kpa)
700	4	1.128	700	6	0.935
1400	6	1.085	1400	8	0.958
2100	6	0.989	2100	7	0.947
2800	5	0.912	2800	6	0.955
3500	7	0.977	3500	7	0.955
4000	6	0.904	4000	7	0.911

A follow up test was performed with the same hydrogel sample in a different pressure sensor because the sample lost a small amount of sensitivity at the end of the initial 100 cycle test. This test was used to determine whether the loss of magnitude of the response was due a change in the hydrogel or in the sensor. The results of this test show that the average magnitude response of this test is 1.4 KPa for swelling with a response time of 4 minutes. The average deswelling magnitude is 1.2 KPa with a response time of 3 minutes. Furthermore, there was no significant difference between the magnitude of the response at the beginning of the test and the response at the end of the test. This validation experiment confirmed that the irregular response of the first test at 18 months was due to the sensor and not the hydrogel sample.

IV. DISCUSSION

The results indicate that the hydrogel samples are responsive to ionic changes, even after an extended period of time in storage. The data gathered from the first two experiments show only a negligible amount of noise, while the third experiment shows a much higher signal to noise ratio. The same piezoresistive sensor was used in all three experiments. As time progresses, the piezoresistive sensing diaphragm loses stability, which generated the noise during the third experiment and likely the decreased response of the hydrogel.

The hydrogel was conditioned for 3 cycles from 55 mM to 165 mM of PBS. The purpose of the conditioning is to create a controlled environment for the hydrogel to swell and deswell. However, the number of cycles was only arbitrarily chosen. The deswelling response of the hydrogel from the first test continued to have a higher magnitude than the swelling response. In addition, the second and third tests both reveal that this magnitude difference can be overcome and equilibrium can be reached after approximately 25 – 30 cycles. The third test demonstrated the most promising results, illustrating that the hydrogel can be tested for more than 40 cycles with consistent magnitudes of swelling and deswelling; however, this only occurred after the initial 25 – 30 cycles. The stable region of the 100 cycle test after 18 months is magnified in Figure 1.9.

a) Storage Stability

After synthesis, the hydrogel monolith was stored at room temperature in PBS solution. The solution was not changed and the hydrogel was stored in natural ambient light. Samples taken from the monolith were within 1 mm of the previous sample taken. This was done in order to obtain results from a homogeneous sample. The data gathered from each test indicate that the hydrogel is able to respond after extended periods of time in storage. This suggests that the shelf life under ambient conditions is greater than 18 months. A figure comparing the response times and magnitudes at the different time intervals is given in Figure 1.10.

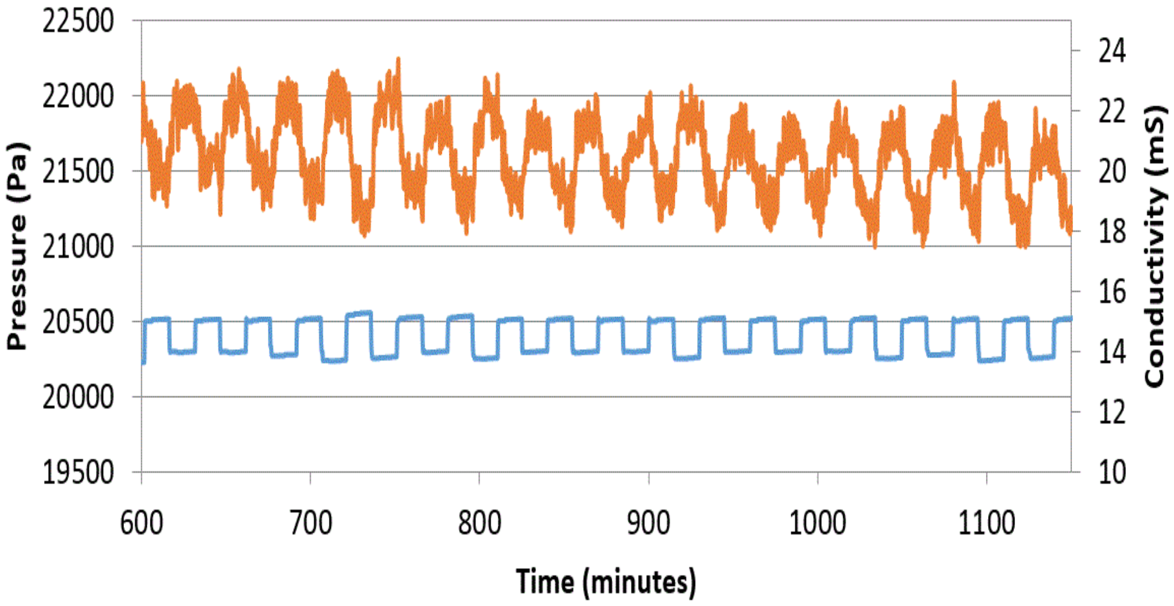

Figure 1.9 : A stable region of the 300 cycle test shows 18 cycles of the third test that illustrate the stability of the hydrogel swelling and deswelling

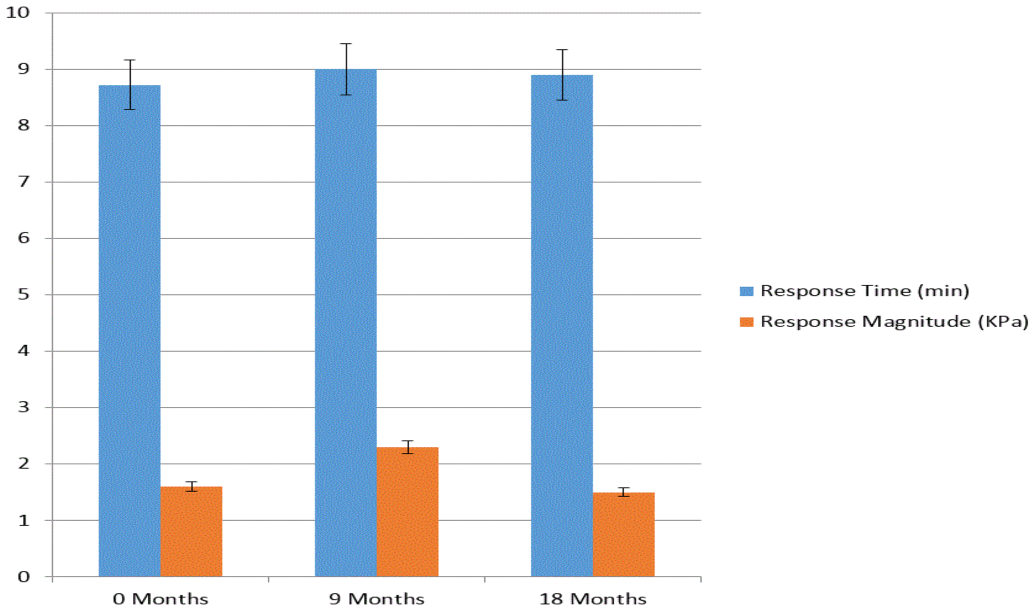

Figure 1.10 : A comparison of the response stability

b) Transportation Tests

The hydrogel samples used in this project were all of the same composition. As discussed in Reference 12, the composition was designed so that the hydrogel swells at low pH. The composition used in this project was designed to have a high sensitivity, and therefore has a higher concentration of DMA than compositions used in other studies [12]. The experiment for the transportation test was designed to determine the effect of response time and magnitude on chemomechanical sensors after experiencing vibrations and exposure to uncontrolled temperatures. The simulated conditions provided evidence that the sensor could be used for further experimentation.

The sensor data gathered from the simulated transportation experiment show that the hydrogel maintains a response to changes in environmental stimuli after transportation, though changes do occur. The data from the control experiment demonstrate a lower magnitude response, 6 KPa compared to 19 KPa after the transportation test, and a longer response time, 75 hours compared to 35 hours. When the hydrogel monolith is synthesized, there are differences in the optical properties across the monolith. The synthesized monolithic hydrogel was not homogenous, and the cross-link density of the hydrogel decreased after simulated transportation. Variation in UV intensity during photocross-linking may also have had an effect. While the two experiments differ in their results, the data demonstrate that the hydrogel maintains its response to changes in environmental stimuli.

c) Operational Stability

There are several factors that may influence the response time and magnitude of a hydrogel sample.

The data gathered during the four experiments demonstrate that the hydrogel has the ability to respond to continuous cycles. The magnitude and time of the responses during each of the tests varied slightly from test to test, but the group of tests shows that the hydrogel will maintain a measurable response to repeated testing. The magnitude and time of the response for the last 20 cycles of the 100 cycles test began to decrease. In order to determine if this was a loss of mechanical properties, a second test was performed on the same hydrogel sample used in that test. The hydrogel was tested in a different sensor for an additional series of cycles to determine the response of the hydrogel after that time. The response time and magnitude remained constant through the follow-up test. The data gathered in the second experiment have helped determine that the decrease in the response was due to a problem with the sensor rather than a loss of response due to the swelling and deswelling behavior of the hydrogel.

V. CONCLUSIONS

The experiments performed in this project were designed to determine if a hydrogel sample could be stored for an extended period of time and to determine if a hydrogel sample could be tested continuously. Samples taken from a hydrogel monolith were tested immediately after synthesis and after 9 and 18 months of storage at ambient conditions. The hydrogel responded in the same manner for all three of the tests.

The experimental results obtained in this project demonstrate that hydrogels can be synthesized, dried, and then rehydrated after a period of time without losing their ability to respond to environmental stimuli. The hydrogel samples were also tested continuously through repeated cycles to determine the effects of the hydrogel after prolonged testing. The hydrogel responded with a similar magnitude and response time throughout the continuous testing with no significant decrease in sensitivity. The results of these tests demonstrate that hydrogels can be used after being stored for an extended period of time, can withstand the stresses of shipping and can be used in continuous cycle testing.

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Novel Aspects of Domino Reaction of Indoles with Homo-Phthalaldehyde and Tere-Phthalaldehyde

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Abstract- In the present work domino reaction of indole with homophthaladehyde (1) have been investigated for the first time. The reaction have been done in presence of glacial acetic acid as catalyst and solvent afford the novel un probable mixture of two isomers A and B of benzo[7]anulene (2) that couldn't be divided accompany with the formation of the predictable tetraindole (3) in a yield of 46 to 38 % respectively. Terephthaladehyde (4) condensed with indoles in glacial acetic acid in a molar ratio (1:4) affording compounds $5_{a,b}$ in a high yield of 93 - 95 % after a short time of stirring at room temperature. Compounds $5_{a,b}$ can act as nucleophile due to the unoccupied two positions of the four indole rings. Accordingly we now present for the first time a expedient method for the synthesis of the novel extensive ring systems 6 and ($7_{a\text{-}f}$) via condensation reaction of compound 5_a with aryl or heteroaryl substituted aldehydes in a molar ratio (1:2).

GJSFR-B Classification : FOR Code: 030601p

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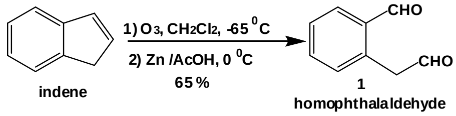
I. INTRODUCTION

A straightforward and direct method for the electrophilic substitution reaction of indole involves the condensation reaction with aliphatic or aromatic aldehydes. Typically these reactions arise in presence of several types of catalysts for instance protic[1-4] or Lewis acids[5-9] or ionic liquids.[10] The electron rich indole nucleus shows an superior reactivity in the direction of carbon electrophiles that commonly results in the formation of three substituted indole derivatives.[11] The 3-position of the indole ring is the favourite site for the electrophilic substitution reactions. 3-Alkyl or 3-acyl indoles are versatile intermediates for the synthesis of a extensive series of indole derivatives possessed pharmaceutical significance.[12]

II. RESULT AND DISCUSSION

a) Synthesis of Homophthalaldehyde

Homophthalaldehyde is of significant concern as a precursor of isoquinoline and its derivatives.[13-17] Homophthalaldehyde (1) is not commercially accessible so that we were synthesized it via an ozonolysis of indene in dry dichloromethane at - 65 ^{0}C followed by reduction with zinc in acetic acid at 0 ^{0}C affording the homophthalaldehyde after an azeotropic distillation to remove the water, [18] (Scheme 1).



Scheme 1: Synthesis of homophthalaldehyde.

After an azeotropic distillation the formed dialdehyde was used straightforwardly in the condensation reaction with indole in acetic acid at room temperature acquiescent a novel benzo[7]annulene derivatives of type 2 in a average yield of 46 % and the tetraindole product 3 in 38 % yield. Both products 2 and 3 are novel compounds which have not reported in the literature.

Compound 2 was set up to be a mixture consists of two isomers A and B, where its ^{1}H-NMR spectrum showed two signals multiplets for the two protons at C_3 of both isomer A and B at δ = 4.98 ppm and 5.71 ppm values, and two signal multiplets for the two protons at C_7 of both isomers A and B at δ = 5.86 and 6.03 ppm. Also the CH_2 group at position 4 in isomer A and at position 6 in isomer B gives each one multiple signal at 2.85 - 2.89 ppm and 3.94 - 4.03 ppm values. These ^{1}H-NMR data give strong suggestion that we have a mixture of both isomers A and B. From the ^{1}H-NMR spectra and referring to the successful single crystal X-ray crystallography of the triindole product that has synthesized and established in our previous work.[19] In which confirmed that the presence of two different signals for the two protons of C_3 and C_7 in every isomer of compound 2, pointed to that we have cis-forms for both isomers A and B, (Scheme 1).

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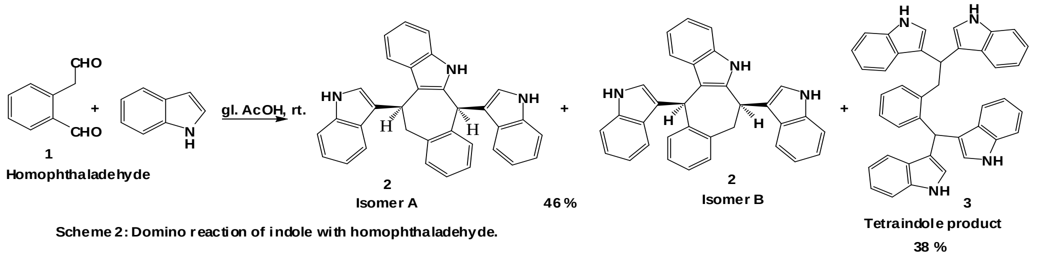
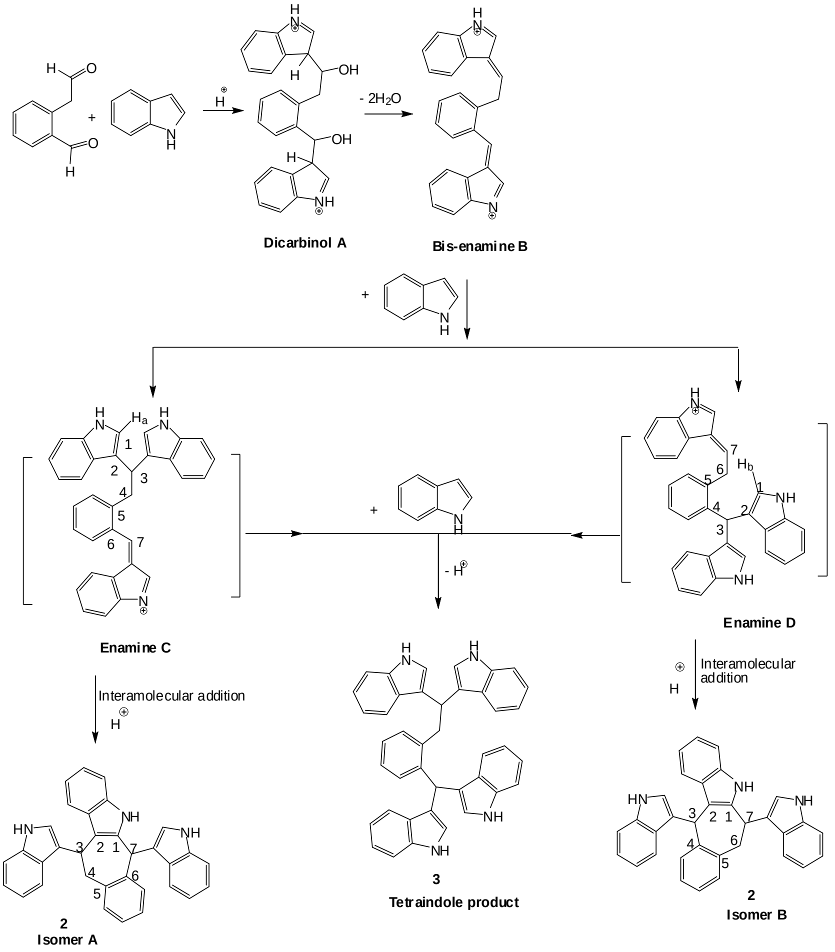


Scheme 2: Domino reaction of indole with homophthaladehyde.

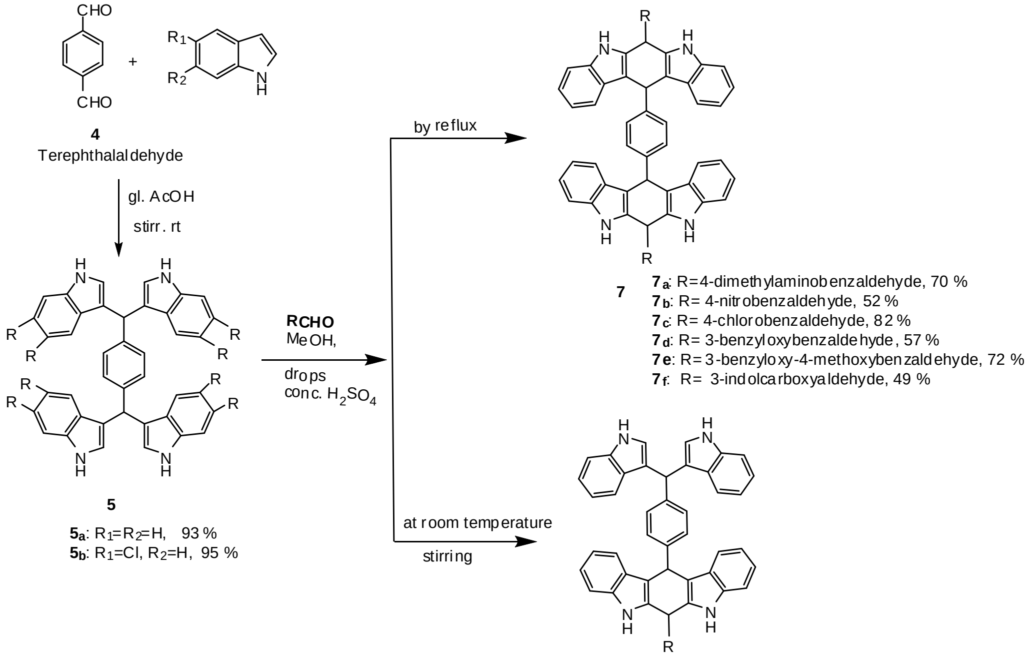


Scheme 3: Expected reaction mechanism for the condensation of indole with homophthaladehyde.

The mechanism of formation of both isomers A and B of triindole (2) accompanied with tetraindole (3) can be explained by the mechanism showed in (Scheme 3). Indole condensed with homophthalaldehyde in acid medium formed the dicarbinol A which underwent dehydration via an elimination of two molecules of water to give the bisenamine B. This bisenamine B reacted with the third indole to give the mono enamine C or D. If the intramolecular addition takes place via α-indole C-Ha in enamine C, the formed triindole (2) was isomer A. And if The intramolecular addition with α-indole C-H_b in enamine D yielded the triindole *isomer* B. A successful addition of the fourth indole to either enamine C or enamine D tends to the formation of the tetraindole product 3. The α-electrophilic attacks in the indole units leading to cyclization have been reported in several indole reactions yielding indole alkaloids.[20,21]

b) Reaction with Terephthalaldehyde

Indolo-carbazoles s have been reported as a primary compound for the synthesis of various drugs and possess important biological, pharmacological, and medicinal activities.[22-24] Indolo-carbazoles are associated with anticancer, antimicrobial and antifungal activities. In most of these belongings biological activity is simultaneous with indolo-carbazoles contain heteroatom. The biological activity depends on the interaction potential with DNA.[25, 26] The electrophilic substitution reactions of indoles with terephthaladehyde have been reported in the literature as a possible way for the synthesis of supramolecular compounds containing bis-indolylmethane (BIMs), namely 3,3$^{/}$,3$^{//}$,3$^{///-}$tetraindolyl(terphthalayl)dime-thane (5$_a$).[27] This reaction has been done in presence of several catalysts such as iodine and N-bromosuccinimide (NBS)28,29 affording the tetra substituted product in good yields[28-32]. In view of the present work, terephthaladehyde (4) condensed with indoles in glacial acetic acid in a molar ratio (1:4) afford compounds 5$_{a,b}$ in a high yield of 93-95 % respectively after a short time of stirring at room temperature 2 to 4 hours, (Scheme 4).



Scheme 4: Condensation reaction of **5$_a$** with aldehydes.

Compounds 5$_{a,b}$ can acts as nucleophile due to the vacant two positions of the four indole rings, hence we now present a convenient method for the synthesis of the novel extended ring systems (7$_{a-f}$) via the condensation reaction of compound 5a with aryl or heteroaryl substituted aldehydes in a molar ratio (1:2), (Scheme 4). The reaction was proficient by let the compounds reacting in methanol solution containing drops of conc. H_2SO_4 under reflux. The product was detected by TLC and isolated easily by column chromatography using dichloromethane as an eluent. However, when the reaction has carried out at room temperature under stirring for long time, the main product which was separated and identified was compound 6 by using p-dimethylaminobenzaldehyde.

Compound 6 can be considered as an intermediate product for the formation of the compound 7$_a$. A modified synthesis for the novel extended ring system indolo-biscarbazoles (7$_{a-f}$) has been done by one step procedure by an acid-catalyzed intermolecular reaction as in the reported belongings of condensation of BIMs and aldehydes to afford the analogous substituted indolo[3,2-b]-bis-carbazoles.[33-37] The proposed mechanism expected that the successful condensation of the tetraindole 5$_a$ with the first mole of aldehyde afforded the bisindole monocarbazole as an intermediate 6 which can be isolated from the reaction.

This intermediate gave the novel extended ring system indolocarbazoles (7) as a final product as a result of the condensation with the second mole of an aldehyde. This suggested mechanism is supported by an isolation of the intermediate 6, so we can say that the second condensation step with the second mole of aldehyde may need heat energy with the acid catalyst for promotion of the reaction to take place. The ^{1}H-NMR spectrum of compounds 7$_{a-f}$ detected the aliphatic hydrogenated protons of dihydroindolo [3,2-*b*]carbazoles as a single signal at δ value of about 4.5 to 5.5 ppm with an integral for four protons and one with an integral for three protons in the case of the compound 6.

III. CONCLUSION

First domino reaction of indole with homophthaladehyde and terephthalaldehyde have been investigated. The reaction have been done in presence of glacial acetic acid as catalyst and solvent affording in case of homophthalaldehyde novel and un expected mixture of two isomers of benzo[7]anulene (2) that couldn't be separated accompanied with the formation of the expected tetraindole (3) in a yield of 46 to 38 % respectively. Terephthaladehyde (4) condensed with indoles in glacial acetic acid in a molar ratio (1:4) affording compounds $5_{a,b}$ in a high yield of 93-95 % followed by condensation with series of aryl aldehydes to synthesis of the novel extended ring systems 6 and $7_{a\text{-}f}$ in a molar ratio (1:2).

IV. Expremental

The melting points were measured on a Boetius-Mikroheiztisch the company "VEB weighing. Rapido Radebeul / VEB NAGEMA "measured and are uncorrected. TLC for the analyzes were with aluminium foil fluorescent indicator from Merck KGaA (silica gel 60 F254, layer thickness 0.2 mm). R_f -values (run level relative to the solvent front). The separations were with column chromatography at atmospheric pressure on silica gel 60 (Grain size from 0.063 to 0.200 mm) from Merck KGaA. NMR spectra were recorded on a "Gemini 2000" (400/100 MHz). The ATR spectra were recorded on a FT-IR spectrometer "IFS 28" by "Bruker. The ESI mass spectra were recorded on a "Finnigan LCQ Classic". The EI mass spectra were recorded on an "Intel 402".

a) *Procedure for the preparation of Homophthalaldehyde (1)*

Ozonolysis of indene: freshly distilled indene (10 ml, 9.8 mg, 85 mmol) was added to dry dichloromethane (200 ml, distilled from P_2O_5). The solution was cooled dawn to -65 ^{0}C, and ozone (about 3 % in oxygen, flow rate 11/min.) was bubbled through the solution for 10 mines. The resulting blue solution was flushed with nitrogen until the blue colour disappeared for about 10 mines, and then zinc (4.0 mg) and acetic acid (20 ml) were added. The solution was allowed to warm to 0^{0}C under stirring. Four similar portions of zinc in acetic acid were added over the next 2 and 1/2 h. The resulting mixture was then filtered, the filtrate washed with aqueous 2 N sodium carbonate solution (50 ml), water (3 x 50 ml), and dried over sodium sulphate. Evaporation of the solvent under reduced pressure gave pale yellow oil. Dry benzene (100 ml) was added and the mixture was heated to distil off the benzene. This isotropic distillation was repeated, and the residue was then distilled under reduced pressure with a yield of 65 % b.p. 90 / 0.1 mm. The distillation temperature must be kept below 100 ^{0}C otherwise extensive decomposition occurs. Colourless liquid, $C_9H_8O_2$, 148.16 g/mol, EI-MS: (m/z) = 148 [M^+] 6 %, 147 [M^+-H] 4 %, 134 [M^+-CH_2] 6 %, 120 [M^+-CO] 97 %, 119 [M^+-CHO] 100 %, ^{1}H-NMR: (400 MHz, DMSO-d_6) δ (ppm) = 4.75 (d, 2H, J=6.5 Hz, CH_2), 7.01 - 7.22 (m, 2H), 7.30 - 7.52 (m, 2H), 9.72 (t, 1H, J=4.7 Hz, CH_2CHO), 10.00 (s, 1H, CHO)

b) *Procedure for the preparation of compounds 2 and 3*

2 Mmol of the aromatic dialdehyde homophthalaldehyde were added in a round bottom flask containing 5 mL of glacial acetic acid at room temperature. Then 5 mmol of indole were added to the reaction mixture. The clear light yellow solution was left to stirring overnight, when the solution became dark brown. The product was detected by TLC (100 % CH_2Cl_2). The TLC showed the formation of the two products, compound 2 at high R_f value and compound 3 at low R_f value where the indole was not finished from the reaction mixture. At this point the reaction was worked up by neutralization with a cold solution of 10 % NaOH affording a brown precipitate. Then the mixture was extracted with CH_2Cl_2 three times 200 mL, washed with water for two times 150 mL and brine for two times 150 mL then dried over anhydrous sodium sulphate, filtered and finally concentrated in vacuum. The crude reaction mixture was purified *via* column chromatography on silica gel eluting with dichloromethane, to remove at first the un reacted indole, and then compound 2 was collected, followed by compound 3.

c) *Cis-distereomer: 6,10-Di(1H-indol-3yl)-5,10,11,12-tetra- hydrodibenzo[a,g]azulene[b]ind- -ole (2)*

Puff powder, $C_{33}H_{25}N_3$, 463.57 g/mol, mp: 255 – 257 ^{0}C, ESI-MS: (m/z) = 462.14 [M^+-H], EI-MS: (m/z) = 463 [M^+] 4 %, 347 [M^+-indolyl] 100 %, 333.9 [M^+-indolyl-CH_2] 13 %, 256.6 [M^+-indolyl-CH_2-Ph] 3 %, 245 [indolyl.CH.indolyl] 9 %, 230 [indolyl.indolyl] 15 %, 217 [indolyl.CH_2.CH.Ph] 12 %, 130 [indolyl.CH_2] 10 %, 117 [indolyl] 20 %, 90 [Ph.CH_2] 8 % , IR-Spectrum: (ATR,cm^{-1}) = 2922 (CH_2), 3401 (NH), ^{1}H-NMR: (400 MHz, acetone-d_6) δ (ppm) = 2.85 - 2.89 (m, 2H, CH_2), 3.94 - 4.03 (m, 2H, CH_2), 4.98 (s,br., 1H, CH), 5.71 (s,br., 1H, CH), 5.85 - 5.89 (m, 1H, CH), 6.03 (s, br., 1H, CH), 6.25 - 6.32 (m, 2H,), 6.22 (s, 1H), 6.68 (s, 1H), 6.85 (t, 2H, J=9.1 Hz), 6.88 - 7.02 (m, 4H), 7.09 - 7.34 (m, 5H), 7.44 - 7.51 (m, 1H), 7.67 (d, 1H, J=7.1 Hz), 7.78 (s, 1H), 10.33 (s, 1H, NH), 10.57 (d, 1H, NH), 10.92 (s, 1H, NH), ^{13}C-NMR: (100 MHz, acetone-d_6) δ (ppm) = 26.32 (CH), 26.34 (CH), 31.47 (CH_2), 32.06 (CH_2), 110.43, 111.17, 111.31, 111.46, 112.73, 116.37, 116.59, 117.34, 117.72, 117.89, 117.97, 118.03, 118.26, 118.49, 119.02, 119.32, 120.23, 120.32, 120.48, 120.69, 123.02, 123.34, 124.77, 125.39, 125.64, 125.90, 126.00, 126.28, 126.33, 126.44, 126.89, 127.73, 128.31, 128.42, 128.89, 131.44, 131.64, 131.89, 134.71, 134.87, 135.13, 135.98, 136.57, 136.73, 138.10, 138.84, 141.51, Elemental analysis: calcd. C, 85.50; H, 5.44; N, 9.06, found C, 85,53, H, 5.42, N.8.99, R_f -Value: 0.72 (CH_2Cl_2), yield: 427 mg, 46 %.

d) *3,3'-((2-(2,2-di(1H-indol-3-yl)ethyl)phenyl)methylene) bis (1H-indole) (3)*

Light brown powder, $C_{41}H_{32}N_4$, 580.72 g/mol, 168 – 172 ^{0}C, ESI-MS: (m/z) = 579.08 [M^+-H], IR (ATR, cm^{-1}): 2922 (CH_2), 3407 (NH), ^{1}H-NMR (400 MHz, DMSO-d_6) δ (ppm): 3.59 (d, 2H, J=6.9 Hz, CH_2), 4.80 - 4.83 (m, 1H, CH-CH_2), 5.71 (s, 1H, CH), 6.13 (s, 2H), 6.47 (s, 2H), 6.72 - 6.77 (m, 4H), 6.89 - 7.19 (m, 8H), 7.22 (d, 4H, J=7.9 Hz), 7.32 (t, 4H, J=7.7 Hz), 10.63 (s, 2H, 2NH), 10.71 (s, 2H, 2NH), ^{13}C-NMR: (100 MHz, DMSO-d_6) δ (ppm) = 41.52 (CH$_2$), 46.00 (CH), 59.01 (CH), 102.00, 105.22, 111.33, 115.02, 116.55, 117.80, 118.03, 118.23, 118.93, 120.44, 122.00, 124.00, 125.50, 126.02, 129.00, 130.00, 132.23, 134.61, 135.02, 138.52, 139.00, 142.00, EA: calcd. C, 84.80; H, 5.55; N, 9.65, found C, 84.87, H, 5.56, N, 9.56, R_f : 0.54 (CH$_2$Cl$_2$), yield: 441 mg, 38 %.

e) *General procedure for the preparation of compound 5$_{a,b}$*

In a flask containing 20 ml glacial acetic acid, (1 mmol, 0.134 gm) of terphthalaldehyde was added under stirring at room temperature. And after all the amounts of the dialdehyde were dissolved, indole (4 mmol, 0.47 gm) or 5-chloroindole (4 mmol, 0.61 gm) was added. Then the reaction mixture was allowed to stirring overnight at room temperature. The reaction solution curved from light yellow to dark pink colour. The product was detected by TLC (100 % CH$_2$Cl$_2$) until the reaction was finished. In the case of indole the product was precipitated from the reaction mixture, filtered off, washed with AcOH, and washed with water until AcOH is removed, dried over P$_2$O5 to give a pure light pink powder of compound 5$_a$,[28,29] in 98 % yield. The case of 5-chloroindole the product precipitated by the addition of 10 ml water and filtered off, washed with water under suction and dried to afford compound 5$_b$

f) *1,4-Bis(bis(5-chloro-1H-indol-3-yl)meth-yl)benzene (5$_b$)*

Pink powder, $C_{40}H_{26}Cl_4N_4$ (704.47) g/mol, mp 160 - 163^{0}C, ESI-MS: (m/z) = 705.35 [M$^+$+H], IR (ATR, cm^{-1}) = 1458 (CCl), 3424 (NH), ^{1}H-NMR: (400 MHz, CDCl$_3$) δ (ppm) = 5.63 (s, 2H, 2CH), 6.49 - 6.50 (m, 4H), 7.05 (dd, 4H, J=1.9, 8.6 Hz), 7.11 (s, 4H), 7.17 (d, 4H, J=7.4 Hz), 7.20 - 7.29 (m, 4H), 7.79 (s, 4H, 4NH), ^{13}C-NMR: (100 MHz, CDCl$_3$) δ (ppm) = 39.67 (2CH), 112.21, 119.15, 119.26, 122.31, 124.97, 125.09, 128.06, 128.71, 135.06, 141.35, EA. calcd. C, 68.20; H, 3.72; Cl, 20.13; N, 7.95, found C, 68.24, H, 3.75, Cl, 20.7, N, 8.00, R_f -Value 0.55 (CH$_2$Cl$_2$), yield (669 mg), 95 %.

g) *General procedure for the preparation of compounds 6 and 7$_{a-f}$*

To a flask containing 50 ml MeOH 1mmol, 0.567 mg of compound 1$_a$ was added under stirring and heating until it completely dissolved. Then 2 mmol of the appropriate aromatic or heterocyclic aldehyde was added to the reaction mixture, and after the aldehyde was dissolved, a few drops of conc. H$_2$SO$_4$ were added drop wisely, where the reaction solution became pink colour. Then the reaction mixture was left stirring for about 1h under reflux. The reaction afforded a precipitate and when the reaction was finished, the precipitate was filtered off while the solution was still hot, dried to afford compound 7$_{a-f}$, which was purified by passing over a column and eluted with 30 % Et.Ac/hexane. Whereas the monocondensed product 6 was formed by leaving the same reactants in a ratio of 1 mmol, 0.567 mg of compound 5$_a$ and 2 mmol, 0.3 gm of 4-*N*,*N*-dimethylaminobenzaldehyde under stirring at room temperature for a long time.

h) *4-(8-(4-(Di(1H-indol-3-yl)methyl)phenyl)-1,2,3,8-tetrahydroindolo[2,3-b]carbazol-2-yl)-N,N dimethylaniline (6)*

Light brown powder, $C_{49}H_{39}N_5$, 697.87 g/mol, mp 218 - 220 ^{0}C, ESI-MS (m/z): 699.12 [M$^+$+H], 697.09 [M$^+$-H], IR (ATR, cm^{-1}): 1590 (N(Me)$_2$), 2850, 2922 (CH), 3407 (NH), ^{1}H-NMR: (400 MHz, acetone-d_6) δ (ppm) = 2.80 (s, 3H, CH$_3$), 2.84 (s, 3H, CH$_3$), 5.93 (s, 2H, 2CH), 6.18 (s, 1H, CH), 6.85 (s, 4H), 6.91 (t, 4H, J=7.3 Hz), 7.10 (t, 6H, J=7.6 Hz), 7.12 - 7.34 (m, 4H), 7.39 (t, 8H, J=8.6 Hz), 9.96 (s, 2H, 2NH), ^{3}C-NMR: (100 MHz, acetone-d_6) δ (ppm): 29.95 (CH$_3$), 30.05 (CH$_3$), 30.26 (CH), 40.86 (CH), 40.93 (CH), 111.90, 112.08, 112.24, 114.01, 118.32, 119.24, 120.10, 120.35, 121.96, 123.15, 123.92, 124.48, 125.18, 128.17, 129.21, 131.82, 133.07, 134.17, 137.33, 143.48, EA calcd. C, 84.33; H, 5.63; N, 10.04, found C, 84.24, H, 5.70, N, 10.00, R_f 0.6 (CH$_2$Cl$_2$), yield (426 mg), 61 %.

i) *4,4'-(8,8'-(1,4-Phenylene)bis(1,2,3,8-tetrahydroindolo[2,3-b]carbazole-8,2-diyl))bis-(N,N-dimethylaniline (7$_a$)*

Pink powder, $C_{58}H_{48}N_6$, 829.04 g/mol, mp 140 - 144 ^{0}C, ESI-MS: (m/z) = 828.26 [M$^+$-H], IR(ATR, cm^{-1}): 1540 (N(Me)$_2$), 3377 (NH), ^{1}H-NMR: (400 MHz, acetone-d_6) δ (ppm) = 2.79 (s, 6H, 2CH$_3$), 2.98 (s, 6H, 2CH$_3$), 5.79 (s, 4H, 4CH), 6.69 (d, 8H, J=8.8 Hz), 6.78 (t, 4H, J=7 Hz), 6.95 (t, 4H, J=6.7 Hz), 7.26 (t, 8H, J=7.3 Hz), 7.62 (d, 4H, J=9 Hz), 9.84 (s, br., 4H, 4NH), ^{13}C-NMR (100 MHz, acetone-d_6) δ (ppm): 26.64 (Me), 39.23 (CH), 40.01 (CH), 111.07, 111.24, 118.39, 118.64, 119.25, 119.50, 121.11, 121.38, 123.48, 123.63, 125.47, 127.31, 128.36,129.36, 129.41, 131.38, 137.06, 137.21, 142.59, R_f 0.44 (100 % CH$_2$Cl$_2$), yield (580 mg), 70 %.

j) *1,4-Bis(2-(4-Nitrophenyl)-1,2,3,8-tetrahydroindolo[2,3-b]carbazol-8-yl)benzene (7$_b$)*

Yellow powder, $C_{54}H_{36}N_6O_4$, 832.90 g/mol, mp 260 – 262 ^{0}C, ESI-MS (m/z): 832.43 [M$^+$-H], IR (ATR, cm^{-1}): 1310, 1516 (NO$_2$), 2852, 2921 (CH), 3406 (NH), ^{1}H-NMR (400 MHz, DMSO-d_6) δ (ppm): 6.02 (s, 4H, 4CH), 6.86 -6.97 (m, 8H), 7.04 (t, 4H, J=7.5 Hz), 7.27 (d, 4H, J=8 Hz), 7.35 (d, 4H, J=8.3 Hz), 7.59 (d, 4H, J=8.6 Hz, 8.15 (d, 4H, J=8.7 Hz), 10.91 (s, br., 4H, 4NH), ^{13}C-NMR: (100 MHz, DMSO-d_6) δ (ppm) = 29.46 (CH), 30.87 (CH), 108.32, 110.33, 111.05, 111.92, 112.06, 112.50, 115.20, 116.14, 117.15, 117.82, 118.34, 118.89, 119.38, 121.02, 121.57, 122.00, 123.89, 124.33, 126.84, 128.50, 129.93, 137.06, 144.22, 146.00, 147.90, EA calcd. C, 77.87; H, 4.36; N, 10.09, found C, 78.00, H, 4.39, N, 10.12, R_f 0.77 (CH$_2$Cl$_2$), yield (433 mg), 52 %.

k) *1,4-Bis(2-(4-Chlorophenyl)-1,2,3,8-tetrahydroindolo[2,3-b]carbazol-8-yl)benzene (7_c)*

Yellow powder, $C_{54}H_{36}Cl_2N_4$, 811.80 g/mol, mp >350 °C, ESI-MS: (m/z) = 811.06 [M^+-H], IR (ATR, cm^{-1}): 2863, 2922 (CH), 3410 (NH), ^{1}H-NMR: (100 MHz, acetone-d_6) δ (ppm): 5.68 (s, 4H, 4CH), 6.77 (s, 4H), 6.82 (t, 4H, J= 4.1 Hz), 6.86 - 6.91 (m, 4H), 6.95 - 7.11 (m, 4H), 7.14 - 7.25 (m, 2H), 7.27 - 7.30 (m, 4H), 7.39 - 7.46 (m, 2H), 7.62 - 7.73 (m, 4H), 9.92 (s, br., 4H, 4NH), ^{13}C-NMR: (100 MHz, acetone-d_6) δ (ppm) = 29.71 (CH), 31.77 (CH), 111.24, 111.99, 114.50, 115.40, 117.34, 118.62, 119.37, 121.11, 121.87, 122.40, 124.39, 125.33, 126.14, 127.55, 128.37, 129.12, 129.92, 130.01, 131.13, 132.50, 134.00, 136.62, 137.21, 137.97, 144.15, EA calcd. C, 79.89, H, 4.47, Cl, 8.73, N, 6.90, found C, 80.02, H, 4.51, Cl, 8.75, N, 6.93, R_f 0.74 (CH_2Cl_2), yield (666 mg), 82 %.

l) *1,4-Bis(2-(3-(benzyloxy)phenyl)-1,2,3,8-tetrahydroindolo[2,3-b]carbazol-8-yl)benzene (7_d)*

Yellow powder, $C_{68}H_{50}N_4O_2$, 955.15 g/mol, mp >350 °C, ESI-MS (m/z): 954.23 [M^+-H], IR (ATR, cm^{-1}): 1456 (C-O), 2922 (CH_2), 3414 (NH), ^{1}H-NMR: (400 MHz, DMSO-d_6) δ (ppm) = 5.05 (s, 4H, 2CH_2), 5.77 (s, 4H, 4CH), 5.95 - 6.08 (m, 4H), 6.43 - 6.77 (m, 8H), 6.82 - 6.85 (m, 4H), 6.92 - 6.95 (m, 6H), 7.03 - 7.09 (m, 4H), 7.11 - 7.24 (m, 8H), 7.34 - 7.41 (m, 4H), 7.51 (s, 1H), 10.68 (s, br., 4H, 4NH), R_f 0.75 (CH_2Cl_2), yield (544 mg), 57 %.

m) *1,4-Bis(2-(3-(benzyloxy)-4-methoxyphenyl)-1,2,3,8-tetrahydroindolo[2,3-b]carbazol-8-yl)benz- ene (7_e)*

Yellow powder, $C_{70}H_{54}N_4O_4$, 1015.20 g/mol, mp 152 - 158 °C, ESI-MS: 1014.25 [M^+-H], IR(ATR, cm^{-1}): 1453 (C-O), 2922 (CH_2), 3391 (NH), ^{1}H-NMR (400 MHz, DMSO-d_6) δ (ppm): 3.69 (s, 3H, OMe), 3.94 (s, 3H, OMe), 5.01 (s, 4H, 2CH_2), 5.58 (s, 4H, 4CH), 6.66 (d, 4H, J=8.51 Hz), 6.76 - 6.71 (m, 6H), 6.86 - 6.93 (m, 6H), 6.97 - 7.18 (m, 4H), 7.21 (t, 6H, J=7.2 Hz), 7.26 - 7.38 (m, 4H), 7.41 (d, 4H, J=7.9 Hz), 7.52 (s, 2H), 10.55 (s, 4H, 4NH), ^{13}C-NMR (100 MHz, DMSO-d_6) δ (ppm): 48.40 (CH), 55.38 (OMe), 55.53 (OMe), 69.83 (CH_2-O), 109.32, 109.58, 110.79, 111.17, 112.04, 112.57, 114.55, 117.92, 118.38, 119.16, 120.26, 120.64, 122.13, 123.23, 123.89, 124.76, 125.66, 126.44, 127.53, 127.75, 128.07, 128.18, 129.12, 136.42, 136.87, 137.02, 141.81, 147.19, 147.61, 148.14, 148.52, R_f 0.69 (CH_2Cl_2), yield (731 mg), 72 %.

n) *1,4-Bis(2-(1H-indol-3-yl)-1,2,3,8-tetrahydroindolo[2,3-b]carbazol-8-yl)-benzene (7_f)*

Light brown powder, $C_{58}H_{40}N_6$, 820.98 g/mol, mp 115 - 118 °C, ESI-MS: (m/z) = 820.25 [M^+-H], IR (ATR, cm^{-1}): 2853, 2923 (CH), 3391 (NH), ^{1}H-NMR (400 MHz, acetone-d_6) δ (ppm): 4.21 (s, 4H, 4CH), 6.94 (t, 8H, J=7.4 Hz), 6.99 (s, 2H), 7.05 (d, 4H, J=7.7 Hz), 7.08 (d, 6H, J=6.9 Hz), 7.35 (d, 4H, J=8.1 Hz), 7.55 (d, 6H, J=8.1 Hz), 9.86 (s,br., 4H, 4NH), 9.96 (s,br., 2H, 2NH), ^{13}C-NMR (100 MHz, acetone-d_6) δ (ppm): 30.26 (CH), 111.27, 111.97, 112.02 (3C), 115.83 (2C), 119.16 (3C), 119.36, 119,67 (3C), 121.89 (3C), 123.28, 123.44 (3C), 128.64, 137.87, EA calcd: C, 84.85; H, 4.91; N, 10.24, found C, 84.88, H, 5.01, N, 10.22, R_f 0.76 (CH_2Cl_2), yield (402 mg), 49 %.

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Physical Properties of Polymer Filled with Inorganic (Granite) Filler

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Keywords: granite, polymer composites, physical properties of composites.

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Physical Properties of Polymer Filled with Inorganic (Granite) Filler

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I. INTRODUCTION

Polymer composites filled with inorganic filler are of interest for many fields of engineering. There is a constant demand for materials engineers to develop better mechanical, thermal and electrical insulation systems that can operate at higher temperatures and greater electrical stress and that can be made for significantly lower cost and higher efficiency[1]. Epoxy composites have been widely used in both the power industry and the microelecronics industry because of their generally superior electrical, mechanical and thermal properties along with their economical and convenient processability. Polymers have low thermal conductivity compared with metal and many inorganic materials, to resist transfer of heat by conduction[2]. The properties of these epoxy/inorganic filler composites depend on the nature of the inorganic filler such as its chemical and physical composition, size, shape and dispersion in the epoxy matrix etc [3].The thermal conductivity of composite materials which are represented as a multiphase substance depends upon the thermal conductivity of each face, the proportion of each phase, and the distribution of the phase [4]. It is well known that the interface in a composite has a significant role in influencingthe properties of electrical insulation materials - especially the thermal conductivity[5]. The random nature of the thermal conductivity process brings the temperature gradient and a mean free bath into the expression for the thermal flux[6]. It is know that the transport of heat in nonmetals occurs by phonons or lattice vibration. The thermal resistance is caused by various types of phonon scattering processes: phonon-phonon scattering, boundary scattering and defect or impurity scattering[7]. in order to maximize the thermal conductivity these phonon scattering process must be minimized. The scattering of phonons in composite materials is mainly due to the interfacial thermal barriers[8].

Thermal conductivity and diffusivity are discussed together because they differ only in that the former is descriptive of equilibrium or steady state conditions and the latter describes non equilibrium or transient conditions. Under steady state conditions, the amount of heat transferred is a function of the temperature difference across the material. For transient conditions, however, the amount of the heat the material can store as internal energy must also be considered [9]. Yet Mehmet dogan study the effect of polymer additives on the physical properties of bitumen based composites the results shows decrease in porosity and thermal conductivity with increase in polymer content [10].

II. EXPERMENTALS

a) *Experimental Materials*

The polymer used in this work is epoxy which is commercial adhesive grade at room temperature curable araldite Euxit50 resin K (Epoxy) supplied by the Egyptian swiss chemical industrials Co., with formulated amine hardener in ratio 3:1 for curing. The epoxy resin is a liquid with low viscosity and transparent in color, the specific gravity of it at 20 °C is 1.05 g/cm3. Granite powder was obtained from a locally available granite industry. Granite's composition shows feldspars and quartz minerals and the particle size of the granite powder ranged from 1 to 100 micrometers, granite has a density of 2.6 g/cm3.

b) *Processing of composite*

To prepare the composite samples, a mould of size 150×150×1 mm3 was made from glass. Glass silicon was used for joining frames, and then plastic sheet was placed in the bottom of the mould. The composites were prepared with hand lay-up technique. The epoxy/granite composites were prepared with 5, 10,15 and 20 wt. % filler content. The epoxies consist of two parts, resin and hardener which need to be mixed in 3:1 volumes to forms the epoxy polymer. For preparing composite samples, granite powder was first washed thoroughly with water and then dried at 100 ^{o}C for 2-3 h

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before using it for preparation of composites then a weighted quantity of granite powder was first thoroughly mixed with a measured volume of epoxy resin. Then a half volume of hardener was added and the result mixture was well mixed so as to obtain a uniform. The mixture was poured into the mould. Then it was covered by plastic sheet. The curing time was around 24 hr. at room temperature 23°C. The composite was taken out of the mould in the form of a plate and was cut and machined to produce samples conforming to the thermal conductivity test samples were cut with 85mm of diameter and 1-2 mm of thickness. For thermal test lee,s disc method was used.

III. MESUREMENTS

a) *Thermal conductivity*

Thermal conductivity is defined as the rate at which heat is transferred by conduction through a unit cross sectional area of material when temperature gradient exists perpendicular to the area. The coefficient of thermal conductivity is expressed as the quantity of heat passes through a unit cube of the substance in a given unit of time when the difference in temperature of the two faces is 1 °C. The quantity of heat flow depends upon the thermal conductivity of the material and upon the distance that the heat must flow. This relationship is expressed as:

$$Q = K/X$$

where Q is the quantity of heat flow, K is the thermal conductivity, X is the distance the heat must flow [11]. Thermal conductivity is also related to the specific heat capacity Cp .This relationship expressed as:

$$\alpha = (k)/(Cp.\ \rho)$$

where α is thermal diffusivity, k is the thermal conductivity, Cp is the heat capacity and ρ is the density[12].

b) *Apparent Porosity*

Porosity originates from the voids, which are created within the bulk. These pores are of two kinds; open and closed pores. The open or otherwise known as apparent porosity measures the fraction of void volume to the material volume. The open pores are usually interconnected so that they provide passages through which gases can pass [13]. The following procedure was used in determining the porosity. The samples were kept in the oven at 1100C for 3h to obtain constant weight W1. The samples was then suspended in distilled water and boiled on a hot plate for 30 minutes. After boiling while still in hot water, the water was displaced with cold water, the weight W2 was measured on a digital balance hinged on the tripod stand. The test sample was removed from the water and extra water wiped off from the surface by lightly blotting the sample with wet towel and the weight W3 of the soaked sample suspended in air was measured. The apparent porosity of the sample was determined from the relationship [13].

$Pa=(W3-W1)/(W3-W2)\times 100\%$

Where;

Pa = apparent porosity
W1 = Weight of the sample
W2 = Weight in cold water.
W3 = Weight in air

c) *Density*

The density of the respective samples was determined basically by measuring the mass and the volume by using the beam balance and the measuring cylinder respectively. It is then estimated from the formula given below [14].

Density (g/cm3) = (Mass (gm))/(Volume (c m3))

IV. RESULTS AND DISCUSSIONS

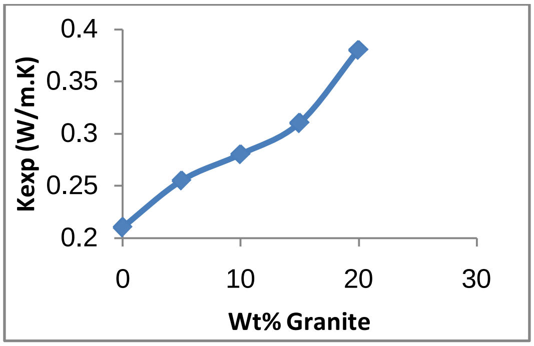

Figure1 : The variation of thermal conductivity Kexp (W/m. K) as a function of granite content

Figure (1) shows that the thermal conductivity increase slightly with increase filler weight fraction .This can be explained because of precipitation of filler particle, which lead to make two layers with more homogenous in distribution, make homogenous media for flowing heat, which make little defect in the material structure lead to gain in energy, and the interface between the matrix and the reinforcement materials was making new path way for heat flow this behavior agree with the obtained results in reference[15].

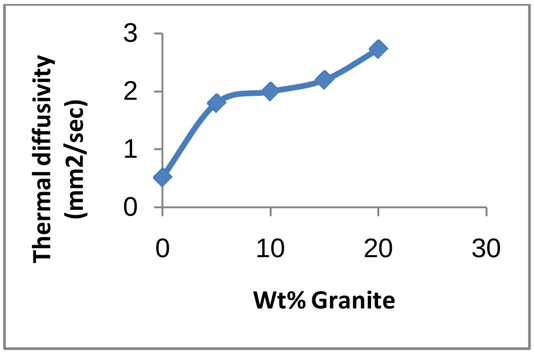

Figure 2 : The variation of thermal diffusivity as a function of granite content

Fig (2) shows that the thermal diffusivity increased with granite content. The increase in granite content cause that the interfacial thermal barriers in composites increase that leads to decrease the phonon scattering and decrease the thermal resistivity that leads to increase the thermal diffusivity This behavior is agreement with[16].

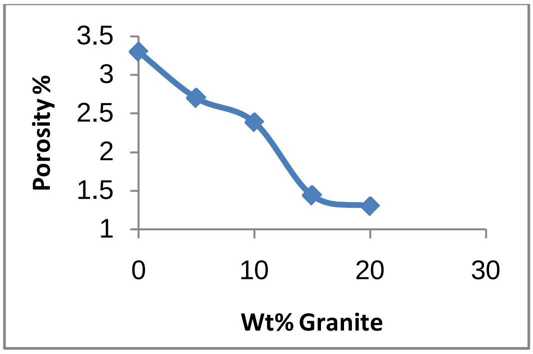

Figure 3 : The variation of apparent porosity as a function of granite content

Voids and air bubbles are a problem in composites. the apparent porosity measures the fraction of void volume to the material volume.

The calculations of porosity give approximate values for void formation during composite preparation. In any case during compression molding of the composites, despite measures taken to reduce air entrapment a small amount of air does get entrapped in the structure.

That reduce void formation leads to decrease the apparent porosity [14, 17].

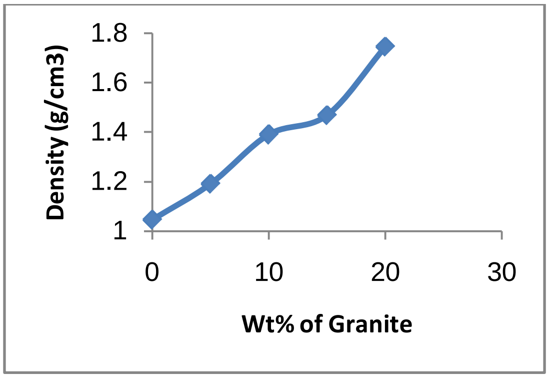

Figure 4 : The variation of density as a function of granite content

Fig (4) shows that the density increase with granite contents this increase in itare normally with the highest density of granite (2.6 gm/cm3) comparable with

Their low density means that they contain fewer atoms per unit volume[17]

V. CONCLUSIONS

1. Thermal conductivity and thermal diffusivity increase with increase of granite weight. This is due to the smaller interfacial thermal barrier at the interface between the particles and the matrix, and the easy pathway among particles.
2. The apparent porosity decrease with increase granite content because of low voids became.
3. The density increase with granite contents this increase in it is normally with the highest density of granite comparable with density of epoxy.

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PCP Removal by Sulfate Radical and Microwave (SR/MW) from Aqueous Solutions

By Ghorban Asgari, Seyed Ali Sajjadi & Afsane Chavoshani
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Abstract- Pentachlorophenol (PCP) is one of the most pollutants frequently used in fungicides and pesticides. Its toxicity is due to intervention with oxidative phosphorylation. Acute and chronic PCP poisoning are approved in human and other living organisms. Recently, AOPs based on microwave and sulfate radical (SO_4^{0-}) have created many great scientific interests due to its high efficiency in the degradation and mineralization of recalcitrant and/or toxic organic pollutants. So that in this study for removal of PCP from wastewater, microwave/persulfate(MW/SPS) was used. The effects of operational parameters such as pH of solution, the power of microwave radiations and the amount of persulfate concentration were studied. Spectrophotometer and HPLC instruments were used for determining the concentration of PCP. The experimental results showed that the removal of PCP was influenced by many factors, such as the pH value, the amount of persulfate and microwave power. The optimum conditions for the best removal rate were obtained at pH=11, a persulfate concentration of 0.02mol/L and microwave irradiation power of 600W for MW/SPS system at constant PCP concentration. Also, obtained results showed that the removal of PCP by MW/SPS and MW alone were follow first order rate decay kinetics and the rate constants were 0.093 and 0.00066 min^{-1}, respectively.

Keywords: pentachlorophenol, oxidation process, microwave, persulfate.

GJSFR-B Classification : FOR Code: 100505, 039999

PCP Removal by Sulfate Radical and Microwave (SR/MW) from Aqueous Solutions

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Abstract- Pentachlorophenol (PCP) is one of the most pollutants frequently used in fungicides and pesticides. Its toxicity is due to intervention with oxidative phosphorylation. Acute and chronic PCP poisoning are approved in human and other living organisms. Recently, AOPs based on microwave and sulfate radical (SO_4^{0-}) have created many great scientific interests due to its high efficiency in the degradation and mineralization of recalcitrant and/or toxic organic pollutants. So that in this study for removal of PCP from wastewater, microwave/persulfate(MW/SPS) was used. The effects of operational parameters such as pH of solution, the power of microwave radiations and the amount of persulfate concentration were studied. Spectrophotometer and HPLC instruments were used for determining the concentration of PCP. The experimental results showed that the removal of PCP was influenced by many factors, such as the pH value, the amount of persulfate and microwave power. The optimum conditions for the best removal rate were obtained at pH=11, a persulfate concentration of 0.02mol/L and microwave irradiation power of 600W for MW/SPS system at constant PCP concentration. Also, obtained results showed that the removal of PCP by MW/SPS and MW alone were follow first order rate decay kinetics and the rate constants were 0.093 and 0.00066 min^{-1}, respectively.

The results of MW/SPS application showed that this processe had suitable efficiency for removal of refractory pollutants and COD and also was cleared that persulfate had high oxidation power.

Keywords: pentachlorophenol, oxidation process, microwave, persulfate.

I. INTRODUCTION

Pentachlorophenol (PCP) is one of the most pollutants frequently used in fungicides and pesticides for wood protection. Its toxicity is due to intervention with oxidative phosphorylation. Acute and chronic poisoning may happen by dermal absorption, inhalation or ingestion. Quantitative determination of PCP in urine and serum is useful to detect its professional or subclinical exposure (Zischke, Arthur et al. 1985, Jorens and Schepens 1993). The clinical features of acute and chronic PCP poisoning can be classified systematically into effects on the skin, metabolism (fever), the hematopoietic tissue, the respiratory system, the central and peripheral nervous system, the kidney and the gastrointestinal tract-(Warwick Fisher 1990, Jorens and Schepens 1993).

Exposure to PCP has also been associated with aplastic anemia, leukemia, other blood disorders and neurotoxicity(Stehly and Hayton 1990, Fisher 1991). Nowadays, environmental researchers have considered the sustainable methods for refractory contaminants removal. The inability of conventional treatment processes to remove PCP has stimulated the development and investigation of advanced oxidation processes (AOPs) based on the formation of high active oxidizing species such as HO^0 and other free radicals. In the last two decades, studies on chemical oxidation by ozone, H_2O_2/UV, Fenton's reaction, photo-Fenton process ,ultrasonic irradiation and TiO_2-mediated heterogeneous photocatalysis suggested the effectiveness of AOPs for phenolic compounds removal(Olmez-Hanci, Arslan-Alaton et al. 2013). Recently, AOPs based on microwave and sulfate radical (SO_4^{0-}) have created great scientific interest due to its high efficiency in the degradation and mineralization of recalcitrant and/or toxic organic pollutants(Liang, Huang et al. 2008, Yang, Wang et al. 2009). Now days, microwave assisted Process has been developed as green chemistry. it is able to reduce or eliminate negative environmental impacts(Anastas and Warner 2000). The use of this system involves the reduction of waste products, non-toxic components, and improved efficiency(Tellez, Alquisira et al. 2011). So that in this study for removal of PCP from Synthetic wastewaters, MW/SPS was used.

II. MATERIALS AND EXPERIMENTAL METHODS

Sodium salt PCP, which is the sodium salt of PCP ($C_6C_{l5}ONa$) purchased from Aldrich (its purity was 98%) and was used without further purification. The characteristics of the PCP included of boiling point: 309-310C^0, mass molar: 288.32g/mol(Fig.1).

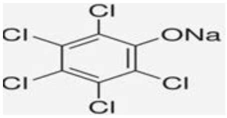

Figure 1 : Chemical structure of PCP

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Under atmospheric pressure, all of the experiments were performed and triplicated in modified domestic microwave oven (2450MHz, M2330 DN, SAMSUNG Co, and output power of 100 to 850 W) (Figure2). Detail modifications of MW were presented as follows: drilled a hole in the upper oven wall and then attached an aluminum tube of the same diameter to the hole then equipped with cooling system and a glass reactor with 500 mL volume. Then Samples were taken at suitable time intervals (10 min) from the reaction reactor with a 10 mL syringe and pipetted in to glass vials. Besides, a Thermometer GENWAY Medal 2003 was utilized to detect variation of solution temperature during degradation process. The leakage of MW oven is measured at 20 cm distance from the aperture.

The PCP solution (100,200,300,400, 500, 750, 1000mg/l) prepared by dissolving PCP in NaOH solution to increase its dissolution solution and adding SPS powders (0.01, 0.02, 0.03, 0.04, 0.05 mol/l) and with adjusted pH (3,7and 11) were put into a reactor. The degradation rate of PCP was calculated according to the following equation:

$$\mu=C_0-C_t / C_0\times 100 \qquad (2)$$

Where:

C_t is the concentration of the PCP after MW irradiation; C_0 is the concentration of the PCP before MW irradiation. PCP was oxidized by the following methods:

a) Microwave irradiation method (MW)

b) Microwave/persulfate method (MW/SPS)

After setting interval time for degradation, samples were drawn for the analysis. Concentration changes of PCP were determined using spectrophotometer according to (APHA, 2005). In the end for identification of oxidation intermediates HPLC spectra and COD methods were used. The measurement with modified HPLC (Part Number.WATO54275 with dimension of 4.6 mm×250 mm and column of symmetry C18-50 μm) method was performed with an acetonitrile/water 60:40 (v/v) as mobile phase at a flow rate of 1mL/min and detection wavelengths of UV was 254 nm. Amount of injected samples was $10\mu l$ at $25C^0$, pH=7 and time of 7minutes. COD was determined using potassium dichromate solution as oxidizer in a strong acid medium, then by titration step using ferrous ammonium sulfate as the reducing agent and Ferroin as the indicator(Hong, Yuan et al. 2012).

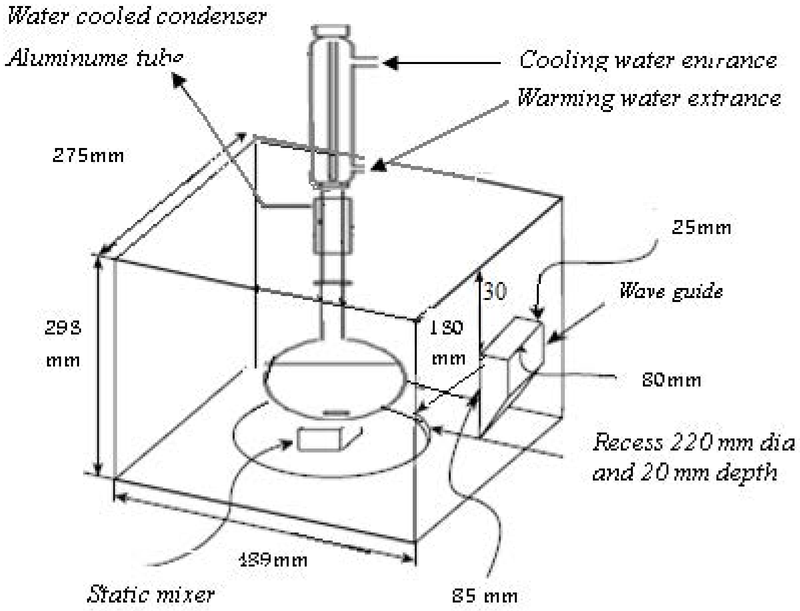

Figure 2 : schematic diagram of modified microwave system

III. Results and Discussion

a) Effect of pH

Degradation of PCP by MW/SPS system in a broad pH range, from acidic to alkaline conditions, was initially studied (Fig3). In our study the values of k in pH= 3,7and 11 was 54%, 64% and 67% respectively. The observed degradation rate was highest at pH 11. The Phenomena of PCP removal are attributed to the ability of MW to initiate sulfate and hydroxyl radical formation at high pH. These radicals at extreme alkaline pH have an higher oxidation potential than acidic conditions .The effect of basicity on persulfate reactivity was discussed by Furman et al(Furman, Teel et al. 2010) and the persulfate can be activated to initiate sulfate radical at high pH, under the effect of hydroxyl radical (Eq. 4)(Ocampo 2009).

$$S_2O_8^{-2} + OH^0 \rightarrow HSO_4^{-0} + SO_4^{0-} + 1/2O_2 \qquad (4)$$

In general and according to experimental conditions following reactions are performed:

All pHs: $SO_4^{0-} + H_2O \rightarrow SO_4^{-2} + OH^0 + H^+$ (5)

Alkaline pH: $SO_4^{0-} + OH^- \rightarrow SO_4^{-2} + OH^0$ (6)

Both SO_4^0- and OH^0 are possibility responsible for degradation of organic contaminants and other radicals may produce depending on pH conditions. other studies showed that in pH=3-10, amount of hydroxyl radical is more than sulfate radical ,But in pH>11amount of sulfate radical is more than hydroxyl(Liang, Wang et al. 2007, Neppolian, Doronila et al. 2010). The difference between our work and previous studies could partly attribute to pH>10. It was also possible that substrates with distinct physico-chemical characterization could show different degradation behaviors in alkaline SPS system. Furthermore, Abu Amr et al reported that a high pH (11) is favored in the removal of Phenol from wastewater using persulfate oxidation(Abu Amr, Aziz et al. 2013). The most of results showed organic removal efficiencies were highly depended on pH values and the efficiencies were dropped sharply with decreasing pH values. For higher pH values (above 11), the efficiencies of removal were relatively high. The complex pH effect may be associated with the fact that the total radical concentration and fractions of the different radicals were varied with pH changes (Gao, Gao et al. 2012).

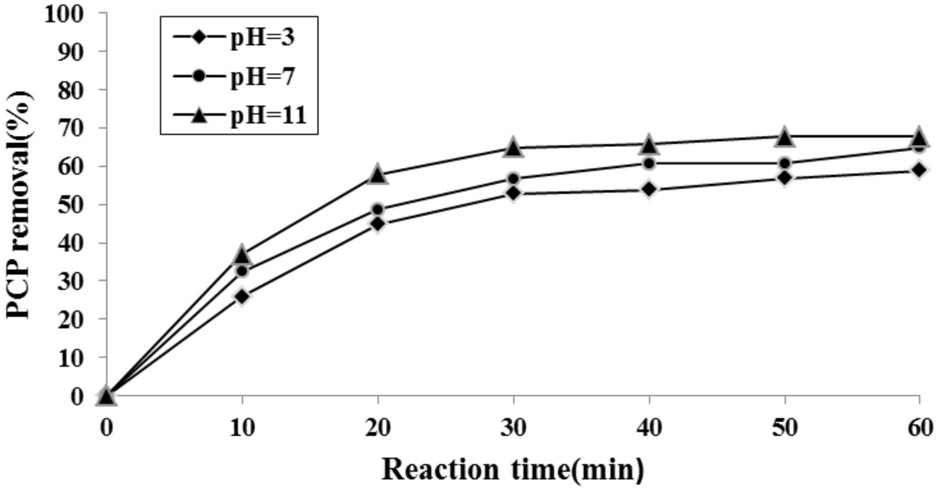

Figure 3 : Effect of pH on PCP removal (C_0=100mg/L, SPS=0.01mol/l, E=600W)

b) *Effect of persulfate concentration*

The effect of the initial persulfate concentration on PCP (100 mg/L) removals was examined at five concentrations of persulfate(0.01,0.02,0.03,0.04,0.05 mol/l) and at 600W. From Fig. 4 it is observed that PCP removal rates increased with increasing persulfate concentration (0.01 to 0.02mol/l). PCP removal was complete after 60 min of reaction. But when increasing the initial persulfate concentration from 0.02 to 0.05 mol/l under MW reaction conditions, increase of persulfate did not effect on enhance PCP degradation. PCP degradation rate almost was decreased 45% when the initial persulfate concentration was increased from 0.02mol/l to 0.05mol/l. An extremely high initial persulfate concentration generated a higher amount of SO_4^{0-} that may reduce PCP oxidation according to the following equations(Olmez-Hanci, Arslan-Alaton et al. 2013):

$$SO_4^{0-} + S_2O_8^{2-} \rightarrow SO_4^{2-} + S_2O_8^{0-} \qquad k = 6.1 \times 10^5 M^{-1}s^{-1} \qquad (7)$$

$$SO_4^{0-} + SO_4^{0-} \rightarrow S_2O_8^{2-} \qquad k = 4.0 \times 10^8 M^{-1}s^{-1} \qquad (8)$$

According to Eqs. (7) and (8), it can be found that condition of extra SPS has a negative effect on $SO4^{0-}$ production, because it changes SO_4^0 radicals trend to anions of SO_4^{2-} and $S_2O_8^{0-}$. Therefore removal efficiency of organic compounds is decreased(Liang, Wang et al. 2007). The decrease in pH values to ≤ 2.5 during examinations ,even by using buffer phosphate, are a consequence of the sulfate radicals formation and their accumulation(Olmez-Hanci, Arslan-Alaton et al. 2013). Also, other researchers confirm these results(Shih, Putra et al. 2012).

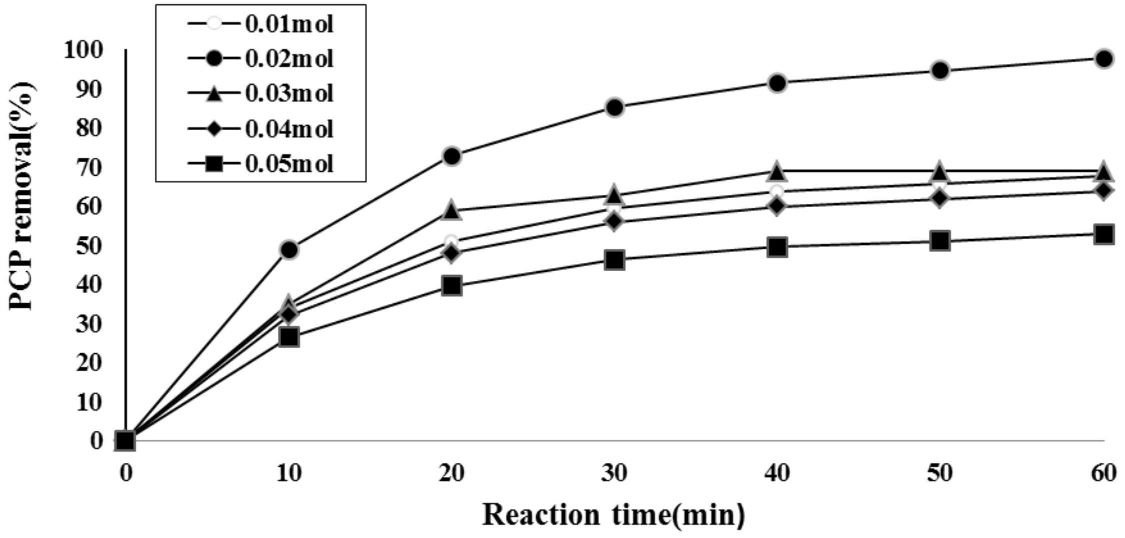

Figure 4 : Effect of SPS dose on PCP removal (C_0=100mg/L, pH=11, E=600W)

c) *Effect of different microwave energy on decomposition of PCP*

The test results shown in Fig. 5 indicated that degradation rate of PCP gradually increased with increasing energy intensity (180,450,600W with optimal temperatures 80, 100, and 105C^0 respectively); whereas the degradation rate did not changed for higher power (>600 W). So the microwave irradiation of 600W was chosen throughout the experiments. According other studies with increasing energy, amount of final temperature (Tf) was increased(Costa, Santos et al. 2009). These results revealed that persulfate could be converted to SO_4^{0-} radicals by MW energy resulting in a considerable enhancement in the reaction rates(Nuechter, Mueller et al. 2003). It was noted that the organic compounds removal by MW/SPS system was higher than conventional heat/SPS (CH/SPS) - system. This effects has been attributed to “specific” or “non-thermal” microwave effects(Tellez, Alquisira et al. 2011) and existence of hot spots. It seems that it be the more possible cause of the reaction rate enhancement observed in works under microwave irradiation. However, it is necessary to more study in this field(Costa, Santos et al. 2009, Hong, Yuan et al. 2012).

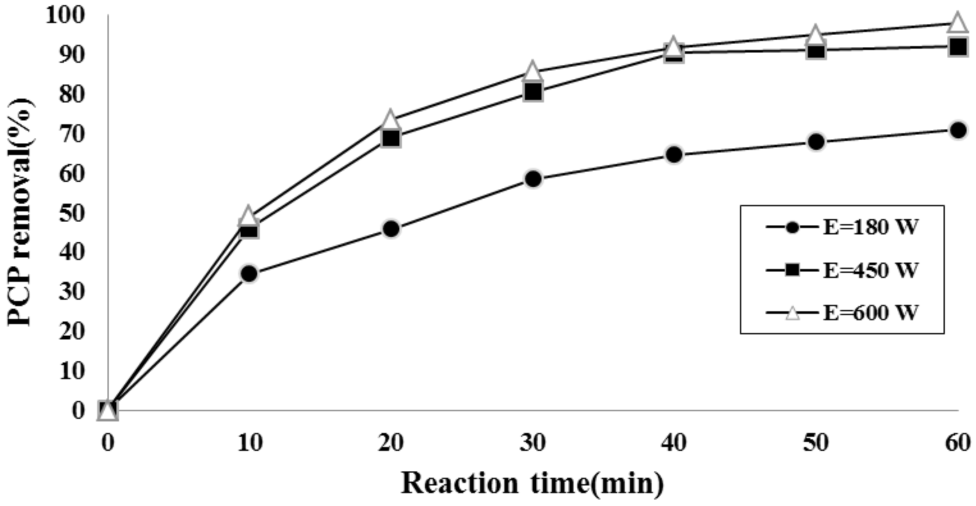

Figure 5 : Effect of energy on PCP removal (C_0=100mg/L, pH=11, SPS=0. 02mol/L, E=180,450, 600W)

d) Effect of PCP concentration

High PCP concentrations were subjected to MW/SPS. In this study effect of PCP concentration (100, 200, 300, 400, 500, 750 and 1000mg/l) was studied by the MW/SPS system under microwave power of 600 W (Fig. 6). In this study the values of PCP removal was between 98%to90%. Because effect of increasing PCP concentration had an ineligible role (8%) on efficiency removal and selection of 1000mg/l PCP wasn't economic, therefore 100mg/l was chosen as optimal concentration. Also, the direct degradation results showed that the removal of PCP was 2% in MW system without SPS after 60 min of MW irradiation. The removal of PCP by MW/SPS and MW alone were follow first order rate decay kinetics and the rate constants were 0.093 and0.00066 min-1, respectively. ¬Today it is observed that processes integrated with MW have high efficiency in removal of pollutants such as phenol, aromatic hydrocarbon and PCPs. For example, this process is able to remove 500 to 12000 mg/l of ammonia with efficiencies above 95% (Lin, Yuan et al. 2009). So that, increasing of no polar organic materials cannot decrease process efficiency. Also other Previous studies confirm that microwave process is very effective for removal of ¬heavy metals, COD and color from waste water effluent (Ramon, Valero et al. 2003, Zhang, Li et al. 2007, Yang, Wang et al. 2009).

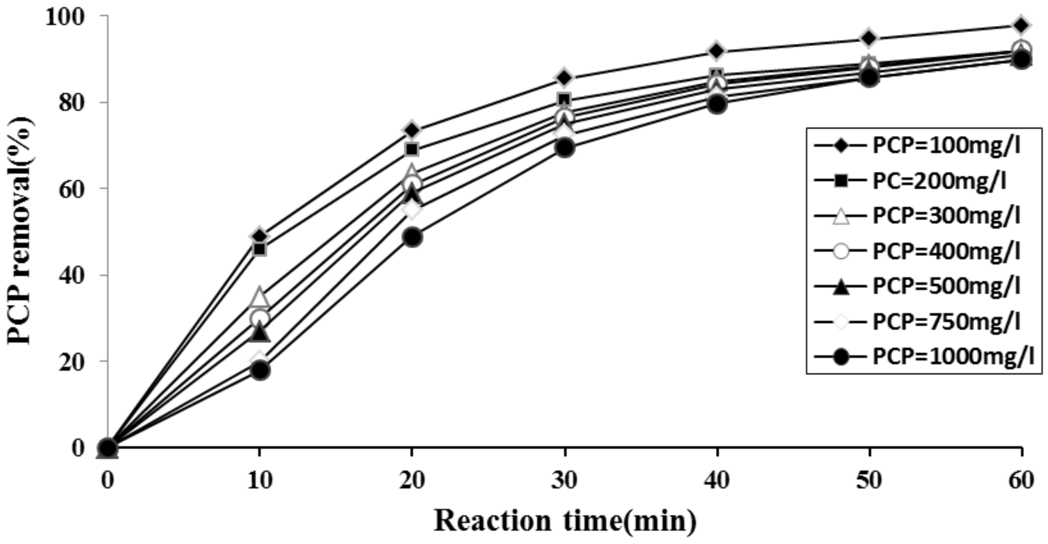

Figure 6 : Effect of PCP concentration (pH=11, SPS=0. 02mol/L, E=600W)

e) Identification of Predominate Radical Species Under Tba Molar Constant

Table 1 shows that the addition of TBA resulted in slightly decreases (i.e., inhibition) in the PCP degradation rate when compared to without TBA conditions. Inhibition was 15% at pH 11. Radical inhibition experiments were conducted to identify the main radical oxidant (SO_4^{-0} vs. OH^0) under 0.04M of TBA by observing the differences in radical reactivates (i.e., radical scavengers. This method has been successfully employed to distinguish SO_4^{0-} vs. OH^0 activity in an activated persulfate system(Anipsitakis, Dionysiou et al. 2006). When comparing k in the presence of alcohol scavengers to those observed without alcohols, the decreases of k due to the addition of the alcohol (% change in k = ((k (with alcohol)/ k (without alcohol)) -1)× 100) were calculated(Liang, Wang et al. 2006) . As shown in Table 1 with the addition of TBA the % change in k (i.e., inhibition) at pH 11 and 0.04 mol/l of TBA was between -31 to -46%. . However, when the TBA was introduced into the degradation systems, the degradation rate was only decreased slightly. This result demonstrated SO_4^{-0} was the dominate active species generated in the MW/SPS system, while OH^0 played a minor role in this process for PCP removal. Usually, SO_4^{0-} is more selective for electron transfer reactions than is OH^0 . So, it is seen that the reactivity of OH^0/TBA system is near 400–1900-fold greater than that of SO_4^{0-} system(Liang, Wang et al. 2007).

Table 1 : Summary of radical identification experiments

Time (min)	K (min^{-1})				% change in K with TBA
	No alcohol	R^2	With TBA	R^2	
10	0.23	97%	0.16	97%	-31
20	0.125		0.059		-32
30	0.058		0.085		-46

f) Comparison of PCP and COD removal

Several mechanisms have been proposed in explaining the organic degradation based AOPs. PCP = 100 mg/L; COD = 344mg/l; pH = 11, $S_2O_8^{2-}$ = 0.02mol/l were analyzed by HPLC and COD method after 0, 10, 20 and 30 min treatment. From Fig 7 it is clear that mainly aromatic intermediates could be detected via HPLC analyses, namely CO_2 and HCL. The reaction degradation can be demonstrated by the products and intermediates shown in the HPLC spectra. COD degradation rate indicated that PCP completely transformed into CO_2 and HCl immediately (Eq.9). Also, The HPLC spectra and COD results showed a similar trend in mineralization and the lack of toxic intermediates and by products in this process.

$$C_6HCL_5O+MW \rightarrow CO_2 + 5HCl \qquad (9)$$

In fact, MW/SPS method provides very good results in efficiency of COD removal and reduces the analysis time from 2 h in standard method to some min for any type of sample(Ramon, Valero et al. 2003).

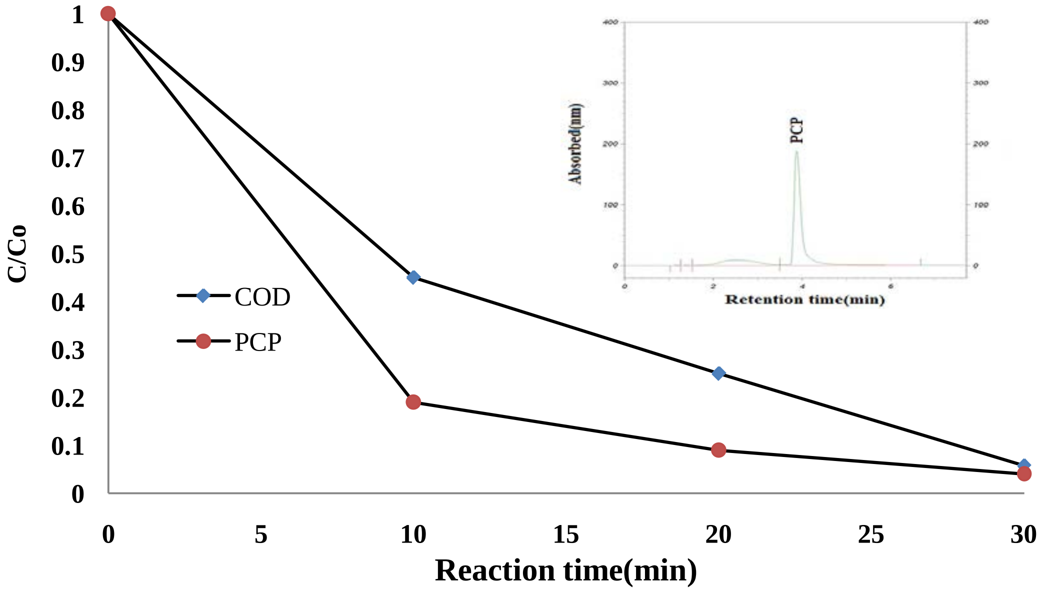

Figure 7 : Comparison of PCP and COD removal in MW/SPS(C_0=100mg/L,COD= 344 mg/l pH=11, SPS=0.-02mol/L, E=600W)

IV. Conclusion

Advanced Oxidation Processes represent a powerful treatment for refractory and/or toxic pollutants wastewaters. Microwave irradiation was particularly effective on COD removal as well as on the oxidation of PCP and eventually leading to mineralization of the compounds into H_2O and CO_2. Addition of SPS, above a required amount, was a limitation for increasing PCP removal. The kinetic analysis showed that a direct reaction of PCP removal with SO_4^{0-} and an indirect reaction with OH^0 radical, because SO_4^{0-} radical was dominant in this process. The removal of PCP by MW/SPS and MW alone were follow first order rate decay kinetics and the rate constants of MW/SPS were 140 times higher than MW alone. This work provides a simple and rapid method for persulfate activation to produce SO_4^{0-} in aqueous solution using microwave energy. As SO_4^{0-} is an active and environment friendly oxidant, it will have great importance in the fields of green chemistry, environmental protection, and other related fields.

V. Acknowledgments

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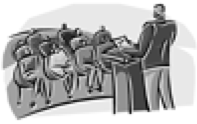

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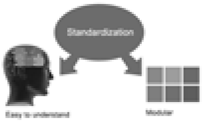

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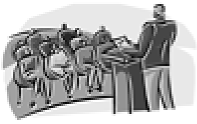

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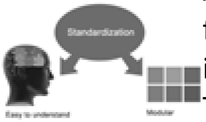

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It is vital, that authors take care in submitting a manuscript that is written in simple language and adheres to published guidelines.

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Standard Usage, Abbreviations, and Units: Spelling and hyphenation should be conventional to The Concise Oxford English Dictionary. Statistics and measurements should at all times be given in figures, e.g. 16 min, except for when the number begins a sentence. When the number does not refer to a unit of measurement it should be spelt in full unless, it is 160 or greater.

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A major linchpin in research work for the writing research paper is the keyword search, which one will employ to find both library and Internet resources.

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Acknowledgements: Please make these as concise as possible.

References

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Abstract	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
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
Methods and Procedures	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
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
Discussion	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
References	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring

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