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## CONTENTS OF THE VOLUME

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- i. Copyright Notice
  - ii. Editorial Board Members
  - iii. Chief Author and Dean
  - iv. Table of Contents
  - v. From the Chief Editor's Desk
  - vi. Research and Review Papers
- 
- 1. Sensitivity of the Computational Domain Aspect Ratio for a Single Rising Bubble in a Hallimond Tube. ***1-4***
  - 2. Cleansing Activity of Polymeric Starch by Reducing the Environmental Hazards to Safe Green Chemistry. ***5-13***
  - 3. Ni-NP/Ni Anodes with Varying Ni: NP Ratio Prepared by Electrodeposition for the Direct Methanol Fuel Cell. ***15-20***
  - 4. Adsorption Kinetics of Sulfates by Anion Exchange Resin Containing Pristine Multiwalled Carbon Nano Tubes. ***21-26***
- 
- vii. Auxiliary Memberships
  - viii. Process of Submission of Research Paper
  - ix. Preferred Author Guidelines
  - x. Index



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## Sensitivity of the Computational Domain Aspect Ratio for a Single Rising Bubble in a Hallimond Tube

By Ashraf Azmi, Periyasamy Balasubramanian, Bawadi Abdullah,  
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**Abstract-** This paper presents how a single rising bubble experiment in the Hallimond Tube can be predicted using computational fluid dynamics model. The study is emphasized on the effect of aspect ratio of cylinder domain to the pressure coefficient and axial velocity around the bubble. A rigid sphere with the radius of 0.00575 m using flow velocity of 0.25 m/s is considered in this study. Numerical and simulated data obtained by other researchers in the similar study were used to validate the simulation results from computational fluid dynamics model. It was observed that a change in width in aspect ratio, causing significant change in the value of simulation data. The highest percentage of difference was observed at the vicinity of  $\theta = 85.26^\circ$  which is 32.4%.

**Keywords:** *aspect ratio; bubble; flotation; grid independent test; hallimond tube.*

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# Sensitivity of the Computational Domain Aspect Ratio for a Single Rising Bubble in a Hallimond Tube

Ashraf Azmi <sup>α</sup>, Periyasamy Balasubramanian <sup>σ</sup>, Bawadi Abdullah <sup>ρ</sup>, Nurul Hasan <sup>ω</sup> & Iylia Idris <sup>¥</sup>

**Abstract** This paper presents how a single rising bubble experiment in the Hallimond Tube can be predicted using computational fluid dynamics model. The study is emphasized on the effect of aspect ratio of cylinder domain to the pressure coefficient and axial velocity around the bubble. A rigid sphere with the radius of 0.00575 m using flow velocity of 0.25 m/s is considered in this study. Numerical and simulated data obtained by other researchers in the similar study were used to validate the simulation results from computational fluid dynamics model. It was observed that a change in width in aspect ratio, causing significant change in the value of simulation data. The highest percentage of difference was observed at the vicinity of  $\theta = 85.26^\circ$  which is 32.4%.

**Keywords:** aspect ratio; bubble; flotation; gridindependent test; hallimond tube.

## I. INTRODUCTION

Flotation is a process of particles capturing by bubbles based on the differences in the physicochemical properties of interfaces [1]. Flotation is widely used by mineral and chemical engineers for the separation and concentration of aqueous suspensions or solutions of a variety of minerals, precipitates, inorganic waste constituents, microorganisms and protein [2-3]. In most of the cases, flotation is used to selectively separate particles from other particles (unwanted).

Bubbles play significant role in flotation process. Therefore, understanding the dynamic characteristics of a rising bubble in water is crucial in flotation process. For this purpose, a laboratory scale flotation device is required. One of such device used for this purpose is Hallimond Tube (HT) as shown in Figure1. HT is a fairly well accepted method for testing

of flotability since it allow researcher to control the mechanical and chemical variables easily [4-5].

Due to shedding of vortices [6], rising bubble experience shape deformation [7-8], helical and zig zag motion [8-10]. This will cause more complications to the study. Therefore, a small rigid sphere is considered in the present work to counter this problem. The computational fluid dynamics (CFD) model is re-create solely based on the HT characterization. Earlier, CFD model was used to study the motion of bubble [11], however the dynamics around a single bubble and effect of aspect ratio (AR) are not available in the literature.

Therefore, a CFD model is created to study the hydrodynamic of a rising bubble in HT. Furthermore, CFD model was implemented using Star-CCM+ V6.04® to determine the dynamics around a single bubble and to investigate the effect of aspect ratio (AR) to the single rising bubble inside HT.

## II. MODEL DESCRIPTION AND TEST CASES

On a single rising bubble in HT study, primary flow was solved. The fluid density was constant at 998.2 kg/m<sup>3</sup> and viscosity was 0.001003 kg/m-s. Bubble rising velocity was 0.25 m/s based on the diameter of spherical solid which is 11.5 mm [12]. Since in this simulation a static spherical solid was used instead of rising bubble, therefore velocity of 0.25 m/s was used as water flow velocity in order to match the phenomena of bubble rising in Hallimond Tube. Location of spherical solid was kept fixed at (0, 0, 0). Figure 2 shows the schematic diagram of the computational domain.

Table 1 : Description of Aspect Ratio Case Study

Aspect Ratio	Test 1 (A1)	Test 2 (A2)	Test 3 (A3)
Width / Length	0.02875 / 0.0575	0.02875 / 0.115	0.046 / 0.0575

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Descriptions of test cases are listed in Table 1. Cell domain AR was used as the case study for this research. The geometries of the domain AR is shown in Figure 3. The AR was calculated on the basis of length from bubble centre to the outlet (L) over diameter of the domain (W). For the model equation, the Navier-Stokes continuous equation and momentum equations for primary flow in cylindrical coordinates were used [13].

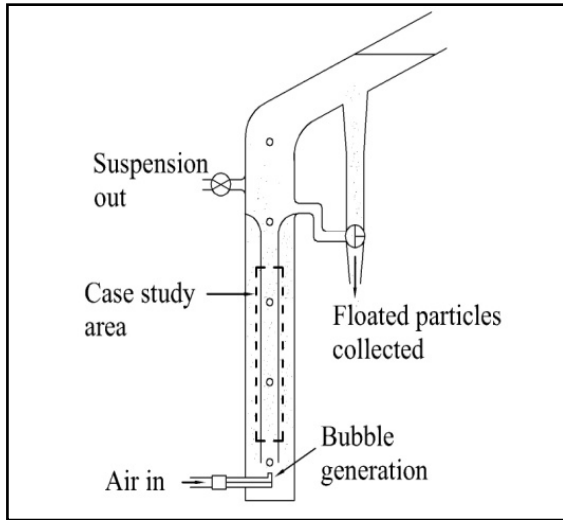


Figure 1 : Schematic diagram of the Hallimond Tube

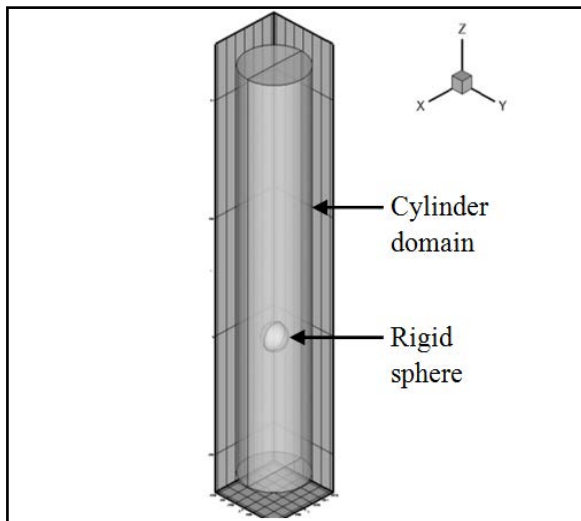


Figure 2 : Schematic diagram of the computational domain

The Navier-Stokes continuous equation in three-dimensional is given by eq. 1

$$\frac{\partial \rho}{\partial t} + \frac{1}{r} \frac{\partial}{\partial r} (\rho r v_r) + \frac{1}{r} \frac{\partial}{\partial \theta} (\rho v_\theta) + \frac{\partial}{\partial z} (\rho v_z) = 0 \quad (1)$$

where,  $\rho$  is fluid density,  $r$  is cylinder radius,  $v_r$ ,  $v_\theta$ , and  $v_z$  are fluid velocity in  $r$ -direction,  $\theta$ -direction,

and  $z$ -direction. The Navier-Stokes momentum balance equation in the  $r$ -direction is given by eq. 2

$$\rho \left( \frac{\partial v_r}{\partial t} + v_r \frac{\partial v_r}{\partial r} + \frac{v_\theta}{r} \frac{\partial v_r}{\partial \theta} + v_z \frac{\partial v_r}{\partial z} - \frac{v_\theta^2}{r} \right) = -\frac{\partial p}{\partial r} + \mu \left[ \frac{\partial}{\partial r} \left( \frac{1}{r} \frac{\partial}{\partial r} (r v_r) \right) + \frac{1}{r^2} \frac{\partial^2 v_r}{\partial \theta^2} + \frac{\partial^2 v_r}{\partial z^2} - \frac{2}{r^2} \frac{\partial v_\theta}{\partial \theta} \right] + \rho g_r \quad (2)$$

where,  $\mu$  is fluid kinematic viscosity,  $g$  is gravitational acceleration. The Navier-Stokes momentum balance equation in the  $\theta$ -direction is given by eq. 3

$$\rho \left( \frac{\partial v_\theta}{\partial t} + v_r \frac{\partial v_\theta}{\partial r} + \frac{v_\theta}{r} \frac{\partial v_\theta}{\partial \theta} + v_z \frac{\partial v_\theta}{\partial z} + \frac{v_r v_\theta}{r} \right) = -\frac{1}{r} \frac{\partial p}{\partial \theta} + \mu \left[ \frac{\partial}{\partial r} \left( \frac{1}{r} \frac{\partial}{\partial r} (r v_\theta) \right) + \frac{1}{r^2} \frac{\partial^2 v_\theta}{\partial \theta^2} + \frac{\partial^2 v_\theta}{\partial z^2} + \frac{2}{r^2} \frac{\partial v_r}{\partial \theta} \right] + \rho g_\theta \quad (3)$$

The Navier-Stokes momentum balance equation in the  $z$ -direction is given by eq. 4

$$\rho \left( \frac{\partial v_z}{\partial t} + v_r \frac{\partial v_z}{\partial r} + \frac{v_\theta}{r} \frac{\partial v_z}{\partial \theta} + v_z \frac{\partial v_z}{\partial z} \right) = -\frac{\partial p}{\partial z} + \mu \left[ \frac{1}{r} \frac{\partial}{\partial r} \left( r \frac{\partial v_z}{\partial r} \right) + \frac{1}{r^2} \frac{\partial^2 v_z}{\partial \theta^2} + \frac{\partial^2 v_z}{\partial z^2} \right] + \rho g_z \quad (4)$$

All the above-mentioned model equations were solved using Star CCM [14].

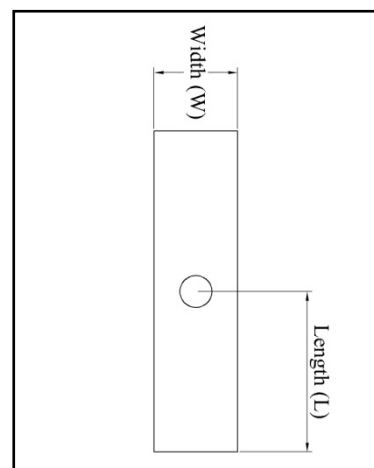


Figure 3 : Aspect ratio (AR), Width / Length

### III. RESULTS AND DISCUSSION

The surface angle  $\theta$ , considered for this study is between  $0^\circ$  to  $180^\circ$ . Numerical simulation data [15] and

simulation data [16] is used for mesh validation. This part of the research is carried out to improve confidence in the CFD prediction on the bubble surface. The results for case validation are shown in Figure 4. Figure 5 shows pressure coefficient,  $C_p$  around a bubble vs. the angular position of the bubble surface.

Assuming the solution is in laminar flow regime under steady state conditions, three refined mesh size around the surface of the spherical solid are used as a part of grid independent test. Grid-independent test is a crucial process in determining the accuracy of the solution [17]. Grid independent solution is obtained for all the meshes. For this study the convergence criteria at  $(10^{-6})$  is sufficient for all meshes, as beyond this there is no further change in  $C_p$ .

A test is performed to study the effect of AR of cylinder to the fluid characterization around the bubble. In this study, three different AR are considered in which the first dimension A1 is used in the case study validation. Radius of the spherical solid, solver parameters such as velocity, courant number, and water density are kept constant. The specification of AR is shown in Figure 3 and the description details are listed in Table 1. For this study,  $C_p$  versus  $\theta$  co-relation is used to study the effect of AR on the fluid dynamics around the bubble surface.

Figure 5 shows the  $C_p$  comparison for flow around the bubble with different computational domain AR. Observing from A1 and A2 plot, it is evident that, length from bubble centre to the outlet (L) does not have a significant effect on the  $C_p$  behaviour. It is also observable that A1 and A2 plot does not show any significant difference starting from the stagnation point ( $\theta = 0^\circ$ ) until they reach the vicinity of ( $\theta = 110^\circ$ ), where  $C_p$  values of A2 start to divert from A1. It is observed that at this angular position, axial velocity,  $v_z$  of A2 is higher than A1.

Comparing A3 plot with the A1 plot, it is observed that the effect of the diameter of the computational domain (W) is relatively significant. Starting from stagnation point, A3 and A1 is having high percentage of difference at 29.14%. The highest percentage of difference is observed

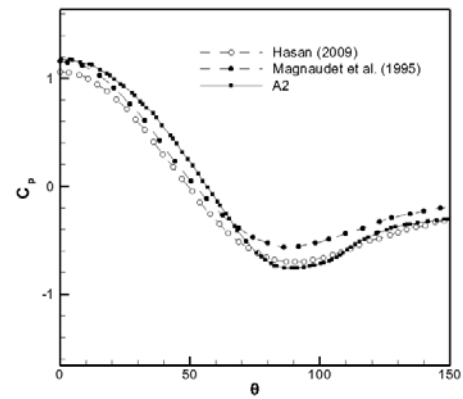


Figure 4 : Validation Test Result.

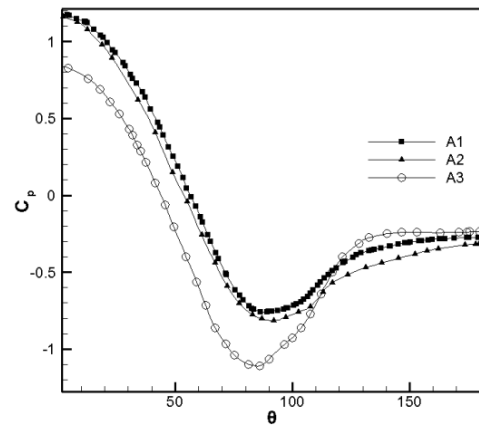


Figure 5 : Profile Comparison of Pressure Coefficient Predictions under Different Dimension of Cell.

Between A3 and A1 at the vicinity of ( $\theta = 85.26^\circ$ ) which is 32.4%. The result is in agreement with the  $C_p$  -  $v_z$  co-relation.

#### IV. CONCLUSION

For the effect of aspect ratio on the fluid characterization around the bubble, 210 divisions around the spherical solid periphery were required to attain grid independent solution. Using a finer mesh offered closer agreement between simulated and numerical results. However, in this study it was observed that using finer mesh causing instability in a solution. In the aspect ratio case study, it was observed that a change in width in aspect ratio, causing significant change in the value of simulation data. The highest percentage of difference is observed at the vicinity of ( $\theta = 85.26^\circ$ ) which is 32.4%. The result is in agreement with the  $C_p$  -  $v_z$  co-relation.

#### V. ACKNOWLEDGEMENTS

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# Cleansing Activity of Polymeric Starch by Reducing the Environmental Hazards to Safe Green Chemistry

By Md. Mohsin Hossain & Md. Ibrahim H. Mondal

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**Abstract-** The bio-degradable starch interaction with various surfactants has been studied for the investigation of ability and cleansing activity of starch-surfactant-water system. The surfactants investigated were sodium dodecyl sulphate, sodium octanoate, cetyltrimethyl ammonium bromide and tween-20. The DS of starch is 0.8 and the concentrations of starch were varied from 0.01 to 1% wt/v. The effect of mixing on the micellisation of the ternary surfactant solutions can be described to a good approximation by taking into account only the effects of the volume difference between the hydrocarbon chains length. Mixed micelle formation with starch depends on the chain-length difference in the same way as for starch-surfactant micelle. Aggregation of the mixed micelles of the surfactants and the polymer coils produced a gel-like complex phase. The water content of the gel phase in equilibrium with aqueous solution increased when the chain-length difference between the two surfactants increased. The more surfaceactive component is strongly enriched in the polymer complexes of gels and it showed maximum cleansing activity of respective detergent. From the experimental results of viscosity, surface tension and other physical properties it indicated that adding starch in detergent as soap filler these properties have changed which also indicated the more cleansing activity of polymeric starch (potato) were cheaply available indoor market in Bangladesh. Biodegradable starch interactions with ionic surfactants by the way of H-bond formation to ensure complexation and reduced the harmful criteria of living organism also to ensure maximum protection of our green chemistry.

**Keywords:** starch, surfactants, cmc, biodegradable, cleansing, XRD and SEM, green chemistry.

**GJRE-C Classification :** FOR Code: 030305, 030203



*Strictly as per the compliance and regulations of :*



# Cleansing Activity of Polymeric Starch by Reducing the Environmental Hazards to Safe Green Chemistry

Md. Mohsin Hossain <sup>α</sup> & Md. Ibrahim H. Mondal <sup>σ</sup>

**Abstract-** The bio-degradable starch interaction with various surfactants has been studied for the investigation of ability and cleansing activity of starch-surfactant-water system. The surfactants investigated were sodium dodecyl sulphate, sodium octanoate, cetyltrimethyl ammonium bromide and tween-20. The DS of starch is 0.8 and the concentrations of starch were varied from 0.01 to 1% wt/v. The effect of mixing on the micellisation of the ternary surfactant solutions can be described to a good approximation by taking into account only the effects of the volume difference between the hydrocarbon chains length. Mixed micelle formation with starch depends on the chain-length difference in the same way as for starch-surfactant micelle. Aggregation of the mixed micelles of the surfactants and the polymer coils produced a gel-like complex phase. The water content of the gel phase in equilibrium with aqueous solution increased when the chain-length difference between the two surfactants increased. The more surface-active component is strongly enriched in the polymer complexes of gels and it showed maximum cleansing activity of respective detergent. From the experimental results of viscosity, surface tension and other physical properties it indicated that adding starch in detergent as soap filler these properties have changed which also indicated the more cleansing activity of polymeric starch (potato) were cheaply available indoor market in Bangladesh. Biodegradable starch interactions with ionic surfactants by the way of H-bond formation to ensure complexation and reduced the harmful criteria of living organism also to ensure maximum protection of our green chemistry. The formed complexes were analyzed and characterized by FTIR spectroscopy, XRD and SEM instrument. Some of the complexes exhibited excellent emulsifying efficiency and surfactants performance properties with this biodegradable starch polymer.

**Keywords:** starch, surfactants, cmc, biodegradable, cleansing, XRD and SEM, green chemistry.

## I. INTRODUCTION

The cleansing activity of soap-detergent is one of the most important phenomena in daily life for the modern society. Thus the improvement of activity of soap-detergent is obviously required for the better quality and performance. Green biodegradable polymers derived from natural resources are potentially very interesting substitutes for non - biodegradable petroleum - based polymers. An attractive field of

application for these polymers is the use as packaging materials. For the current petrochemical based products recycling is often neither practical nor economically feasible [1]. Natural polymers such as starch, cellulose or proteins are potentially very interesting starting materials for biodegradable packaging materials. In particular starch is attractive as it is relatively cheap and abundantly available. However, the use of native starch for packaging materials is limited due to its low moisture resistance, poor processability (high viscosity), high brittleness, and incompatibility with hydrophobic polymers. Further modification of starch is therefore required to introduce hydrophobicity and to improve mechanical and moisture barrier properties. Thus the interactions may be intra and/or intermolecular. The balance depends on the structural parameters of the polymer, such as the nature, lengths and content of hydrophobic groups, their distribution along the starch, the hydration capacity, the degree of polymerization, polymer concentration and on other parameters such as salinity, pH and organic co-solvents [2], [3]. Among the associated polymers, amphiphilic polysaccharides with a natural non-toxic and biodegradable carbohydrates are of particular interest. They were prepared by the hydrophobic modification of a variety of polysaccharides, such as Corn, potato [4], hydroxyethylcellulose, carboxymethyl cellulose [5], [6] and pullulan [7]. Increasing interest has been focused on the structure– solution property relationship of amphiphilic polysaccharides [8], [9], [10] and [11]. The associative behaviour of hydrophobically modified carboxymethyl cellulose and Carboxymethyl pullulan (CM-pullulan) were studied after the amidation of these polysaccharides in DMSO [10], [7]. In previously studied, the hydrophobization of various polysaccharides were investigated, such as hydroxyethylcellulose [12] carboxymethyl cellulose [13], xylan [14], and carboxymethyl starch [15], [16] by the esterification of hydroxyl groups using classical (with acylchloride and mixed anhydride) and unconventional methods.

The interactions of surfactants with cationised cellulose, has been studied by [17], [18] and nonionic cellulose ethers have been subject of extensive studies by [19]. The structure of starch is very similar to cellulose, but the difference on the bindings, which link

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the monoglucose units to form the polymer, makes their chemical behavior very different. The polymer chains in starch are much more flexible than in cellulose, making the polymer more soluble in different solvents. The polymer chains of amylopectin in starch are also branched, whereas cellulose has completely straight chain. Thus, actually starch is very different material to cellulose despite of their chemical similarity.

The Infrared spectra of starch and related compounds have been studied for a long time by a number of authors Rowen and co-worker [20] and [21] studied the infrared spectra in the detection of chemical changes in starch and some other starch-surfactant derivatives and investigated the effect of Hydrogen bonding and change in crystalline structure on the infrared spectrum of starch. Starch-surfactants complex interactions of H- atom of starch within the surfactant molecule by the H-bonding process are now subject to IR absorption of the functional groups which may vary over a wide range.

From the above mentioned features, it is proved that many researchers have paid their attention on this field. In spite of half century of great effort, many academic aspects such as, chemistry, chemical reactions, bond formation on starch-surfactants interaction are still open for discussion. The purpose of the present investigation is to explore the effect of starch interaction with various surfactants and the better understanding the mechanism between starch and surfactants complexes studied by the ternary phase diagram, interfacial surface tension and viscosity measurement and characterized by the XRD and SEM analysis.

## II. EXPERIMENTAL

### a) Materials

Potato starch as powder form was purchased from UNI-CHEM, China and its degree of substitution (DS) was 0.80. Starch solution was prepared by heating the starch in water in an autoclave at 120°C for 30 min.

All solutions were prepared at least 24 h before measurement was performed. The surfactants sodium dodecyl sulphate (SDS), N-cetyl- N,N,N-trimethyl ammonium bromide (CTAB), sodium octanoate (NaOct) and Tween 20 were purchased as analytical grade and were used without further purification. The water used was ion exchanged and distilled. Its conductivity, and reduced viscosity were 2.0  $\mu\text{S}$  and 4.0  $\text{dm}^3/\text{mol}$ , respectively and its surface tension was  $71.5 \times 10^{-3} \pm 0.5 \text{ N/m}$  at 30°C. All other chemicals were analytical grade and were used without further purification.

### b) Methods

#### i. Surface and Interfacial Tension Measurement

Surface tension was measured with a drop weight method (Stalagmometer instruments). In the

calculation of surface tension, the correction factors of Huh and Mason [22] were used. The reproducible result between measurements of the same sample was  $\pm 0.5 \text{ mN/m}$ . The results of the surface tension measurement were presented as ( $\gamma$ ) values calculated from  $\gamma = \frac{mg}{2\pi f}$ .

where, f is equal to  $\frac{1}{v^3}$ , v is the volume of the drop

and r is its radius, mg is the weight of falling drop and  $\gamma$  is its surface tension. A drop of the weight (mg) given by the above equation has been designated as the ideal drop. Repeated measurements (2-4 times) were conducted on each sample from which equilibrium surface or interfacial tension values were obtained by averaging the values at very long periods, where the surface and interfacial tension values showed little or no change with time. Prior to running tests with the starch solutions, the instrument was calibrated with water and then checked by measuring the interfacial tension between water and pure starch.

### c) Viscosity

Viscosities were determined with an Ostwald viscometer according to British standard (Fisher Scientific TM 200) with a fluctuation of  $\pm 0.10 \text{ C}$  was used. The flow of time was recorded by a timer accurate up to  $\pm 0.01$  second. At certain surfactant/starch ratios the aggregates formed were very mobile flocks, which tended to form in the samples. This could be partly avoided by draining the capillary fully between measurements. The results of the viscosity values

calculated from  $\eta_{red} = \frac{(t-t_0)}{t_0} \cdot \frac{C}{c}$ , where t is the measured

efflux time of solutions and  $t_0$  is the efflux time of the pure solvent (water) and C is the weight concentration of the surfactant, starch & surfactants mixed polymer.

### d) Ternary Phase Diagram

For the development of ternary phase diagrams, the sample components were taken into the test tube by varying composition in such a way that the total composition remains 100%. The components were added by varying weight or volumes. The samples were prepared by varying 5% composition of two components simultaneously keeping the third component constant, alternatively in a test tube. The open end of the test tube was then closed with rubber cork in such a way that the vapour would come out and would enter into the test tube, here the cork reacted with the sample. The samples were then shaken for well mixing of the components and placed into the diagram are put according to the composition and mark. After completion of the 228 samples according to the diagram on the wooden frame. The open end of the test tube was then closed tightly with cork, so that this would



remain no leakage or the lower part of the cork did not touch the sample solution and after that these samples were then left to equilibrate in a thermostat box at 30°C for at least ten days and would be shaken from time to time. The equilibrium was established within this period.

#### e) Characterization

##### i. SEM Analysis

Scanning Electron Microscope (SEM, JEOL, JSM 6301F, Japan), Fine coater (JFC 1200, JEOL, Japan), Aluminum specimen stub, Double-sided adhesive tape.

##### ii. Procedure for Sample Determination

Scanning Electron Microscope (SEM) of potato starch, surfactant sample and starch-surfactant complexes samples were less than 4% moisture content before examined. Dried sample and sprinkle were taken onto the double-sided adhesive tape attached to the specimens tub.

The excess sample was removed and the sample was placed in fine coater of gold coating for 150 seconds. The coated sample was then placed in the sample chamber of the SEM (Appendix Fig.7.1). The sample was examined at magnification of 2,500 and 6,000 with the accelerating voltage of 10 Kv.

#### Model and Specification

##### iii. XRD Analysis

##### iv. Preparation of Sample for XRD

The preparation of starch sample, the dried sample is saturated with water by stirring repeatedly with a glass rod; this step is performed by 2g of starch with 100 ml of hot water which is 100% distilled and stirred at room temperature for 1h, centrifuging the suspensions and decanting the supernatant solutions. This process is repeatedly three times. Then the starch solution is dried in oven at temperature 80°C. 2h after proper drying the powder sample is kept in a sealed bottle. The syntheses of cationic surfactants were undertaken by the following procedure: 2g of Sodium dodecyl sulfhate was first dispersed in 100 ml of de ionized water then under mechanical stirring for about 1h. A pre dissolved starch solution of same amount was slowly added to that suspension near about at 80°C. The reaction mixtures were stirred for 1h at 80°C using mechanical stirring after proper drying the powder sample is kept in a sealed bottle then dried products were stored in vacuum desiccators.

Starch powders to be used for X-ray diffraction (XRD) measurements were equilibrated in desiccators containing saturated solutions of  $K_2CO_3$  at 20-22°C. Under these conditions, the relative humidity (RH) at 20°C was shown to be 44% and the final water content of pea, maize, potato, and wheat starches was 13-14, 12, 15, and 12%, respectively. Wet starch powder (from potato) for XRD measurements was produced by first equilibrating the starch in excess water. The starch

suspension was then centrifuged (3000 *g*) and the supernatant removed. The starch precipitate appeared as a hard wet powder that was slightly more moist at the top. This moisture was dried with tissue paper. The wet starch powder had a water content of 49%. It was apparent that the proportion of water was slightly overestimated, because the precipitated starch granules would have a small amount of free space between them, which would be filled with free water. This overestimation, however, can be considered to be very small as the granules in the precipitate were closely packed. Since the water content within the crystallites is fixed, near about 24% the proportion of water in the amorphous part of starch can be estimated at 55- 60%. It was assumed that total crystalline of starch and complexes.

*Apparatus* Wide-angle X-ray diffractometer (JEOL, JDX 3530, Japan), Silicon sample cell and Computer with program MDI Jade 6.5 (Japan).

### III. RESULTS AND DISCUSSIONS

Some of the prepared starch-surfactant mixture lowered the surface tension of water, namely at lower concentration of the sample (Table 1). The functional properties of some of the prepared surfactant and starch mixed surfactant solutions were tested for emulsifying efficiency, washing power and antiredeposition efficiency. The emulsifying efficiency was characterized by the stability of the parafinic Tween-20/water emulsions and other surfactant mixture at definite ratio. The results summarized in Table 2 which show that some of the surfactant made emulsions of the oil/ water type stable even after 24 h. Their efficiency was comparable to that of the commercial emulsifier Tween 20. Some of the tested mixture showed excellent washing power exceeding that of the anionic detergent, name SDS containing dodecyl chains. The antiredepositive efficiency was higher than the starting SDS, but moderate in comparison to starch used as a co-builder in detergents [23].



**Table 1 :** The Value of Surface Tension of Surfactants (Cationic, Anionic and Non-Ionic)) with Added Starch

% Log Conc. of surfactant solution	% Conc. of surfactant solution	Surface tension of SDS mixed with starch soln.	Surface tension of CTAB mixed with starch soln.	Surface tension of Tween-20 mixed with starch soln.
-2.00	0.01	49.11	49.19	53.11
-1.69	0.02	48.02	47.15	50.19
-1.52	0.03	45.35	45.67	49.75
-1.39	0.04	44.31	44.89	48.61
-1.30	0.05	43.13	44.15	46.63
-1.22	0.06	42.95	43.37	45.84
-1.15	0.07	42.73	42.69	45.45
-1.09	0.08	42.55	42.46	45.05
-1.04	0.09	42.52	42.45	44.05
-1.00	0.10	41.51	42.41	44.07

**Table 2 :** At Temperature Ranges (25o-85oc) Solution Viscosity of Starch Mixed Surfactant CTAB

Temp °C	Reduced Viscosity (polymer)					reduced Viscosity (polymer-surfactant mixture)				
	0.0625%	0.125%	0.250%	0.500%	1.000%	0.0625%	0.125%	0.250%	0.50%	1.0%
25	1646.523	810.610	417.380	211.138	118.29	1545.39	910.251	534.50	288.45	181.67
35	1380.073	821.446	471.486	289.594	180.15	1291.59	756.736	414.09	248.74	148.57
45	1214.395	778.756	421.537	248.772	161.83	1171.50	700.102	332.94	226.80	133.51
55	1073.070	640.599	364.620	238.510	130.61	1011.82	603.996	366.08	182.87	124.72
65	920.668	571.382	381.039	208.818	128.11	911.044	546.011	367.44	158.34	103.61
75	847.704	529.308	319.558	184.321	114.53	812.917	468.325	249.96	155.25	98.76
85	746.026	478.272	286.076	168.94	99.96	753.728	441.672	252.48	141.58	89.44

**Table 3 :** At Temperature Ranges (25o-85oc) Solution Viscosity of Starch Mixed Surfactant SDS

Temp °C	Reduced Viscosity (polymer)					reduced Viscosity (polymer-surfactant mixture)				
	0.0625%	0.125%	0.250%	0.500%	1.000%	0.0625%	0.125%	0.250%	0.50%	1.0%
25	1646.523	810.610	417.380	211.138	118.29	1545.39	910.251	534.50	288.45	181.67
35	1380.073	821.446	471.486	289.594	180.15	1291.59	756.736	414.09	248.74	148.57
45	1214.395	778.756	421.537	248.772	161.83	1171.50	700.102	332.94	226.80	133.51
55	1073.070	640.599	364.620	238.510	130.61	1011.82	603.996	366.08	182.87	124.72
65	920.668	571.382	381.039	208.818	128.11	911.044	546.011	367.44	158.34	103.61
75	847.704	529.308	319.558	184.321	114.53	812.917	468.325	249.96	155.25	98.76
85	746.026	478.272	286.076	168.94	99.96	753.728	441.672	252.48	141.58	89.44

**Table 4 :** At Temperature Ranges (25°-85°C) Solution Viscosity of Starch Mixed Surfactant Tween-20

Temp °C	Reduced Viscosity (polymer)					reduced Viscosity (polymer-surfactant mixture)				
	0.0625%	0.125%	0.250%	0.500%	1.000%	0.0625	0.125%	0.250%	0.500%	1.000%
25	102.783	96.837	101.702	114.032	102.783	134.877	106.567	149.427	116.297	171.765
35	86.567	81.162	102.921	98.346	120.374	113.503	144.299	120.367	170.153	157.970
45	76.837	73.594	89.849	87.888	106.633	98.236	123.786	108.401	151.884	135.538
55	66.567	63.864	80.699	76.124	93.656	86.022	109.256	96.435	133.615	123.843
65	57.918	57.378	71.549	67.627	85.259	76.862	100.709	84.470	121.115	107.654
75	53.594	51.432	65.666	61.091	78.389	69.992	92.162	77.632	110.538	98.976
85	48.189	47.108	59.784	55.209	71.519	63.122	84.470	70.794	99.961	87.378

Several references may be found in the literature to the compaction of poly electrolytes on binding of oppositely charged surfactants [24]. Viscosity measurements have been carried out by [25] on mixtures of PSS of Mw of 130 000 and DTAB. When added to solution of 0.36 w% PSS, DTAB in the pre-precipitation zone brought about a progressive reduction in the viscosity of the solutions. For example, 5.8 mM DTAB was found to drop the reduced viscosity of PSS by a factor of ten, far exceeding the reduction effected by addition of the simple analog "surfactant", tetradecyltrimethyl ammonium bromide. The explanation offered was that coiling of the flexible "vinyl" backboned

poly electrolyte occurs around small clusters of the surfactant, which form under these conditions. Similar effects have been obtained by [26] who studied the viscosity characteristics of a series of amphoteric polymers on addition of anionic (SDS) or cationic (CTAB) surfactants to their solutions. [27] and [28] have reported viscosimetric and rheological studies on two cationic polymers, viz. a cationic cellulosic (Polymer JR) and an acrylamide/methacryl oxyethyl trimethylammonium chloride co-polymer (Reten, Hercules) within a range of polymer and added SDS concentrations. Considerable differences in behavior between the two polymers were found.

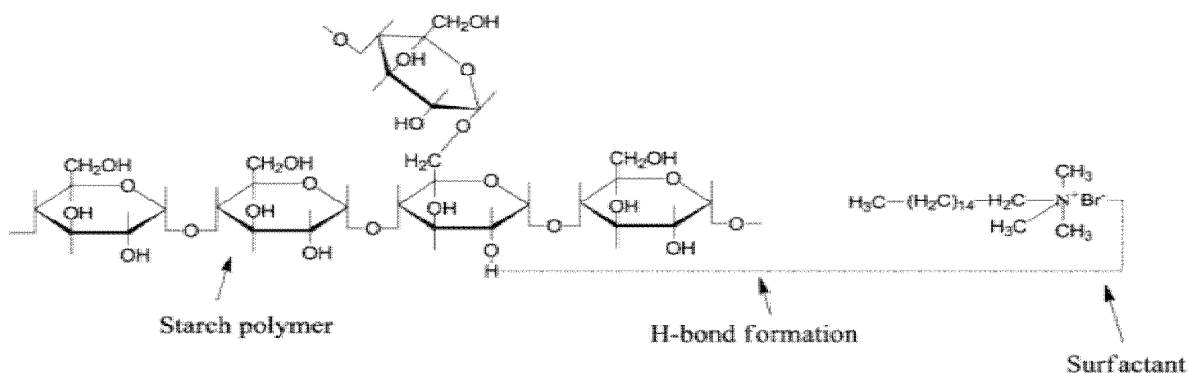


Fig. 1 : H-Bond Formation Starch with Cationic Surfactants

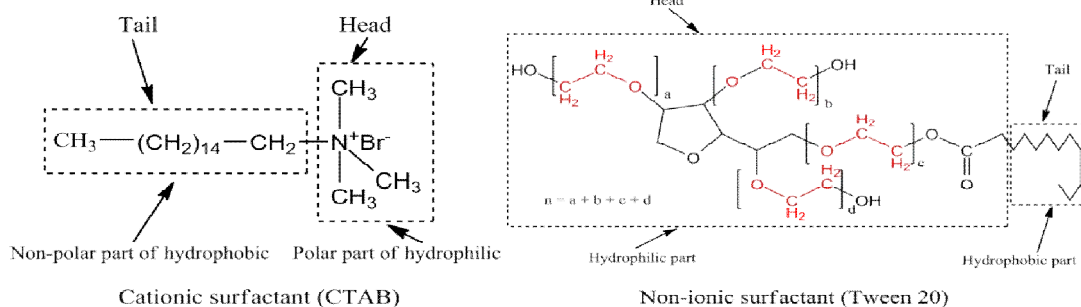


Fig. 1 (a) : Cationic Surfactant Molecule Fig. 1(b) : Structure of Non-Ionic Surfactant Absence of Polar Part

From Fig.1 it has been found starch molecule have many hydroxyl group and hydrogen atom which bind with surfactant molecule through H-atom called H-bond formation. So bond breakdown is easily of hydrophilic and hydrophobic part of surfactant molecule finally cleansing activity increased although starch are biodegradable and eco- friendly. Our motivation for the present study is not only scientific interest. Poly soaps are present in the wash liquor where they might form micro domains. Amphiphilic molecules may bind to these domains, and the concomitant reduction of free surfactant in solution may adversely affect wash performance of surfactants. The Fig.2 (b) of non-ionic surfactant under the region given where it has been

seen that the non ionic Tween -20 molecule is totally dipole absent so, there is no chance to make bond formation through H atom by hydrogen bonding with starch molecule.

From results analysis it has found at lower concentration the reduced viscosity value are high but at CMC point the curve become level off but after increasing concentration the reduced viscosity value increased slowly in all cases but in case of ionic surfactant (SDS, CTAB) the changing is remarkable due to maximum interactions occurred with starch polymer. Here, we mentioned that temperature has a remarkable effect in the complexes of starch-ionic surfactant, here it is obtained according to Arrhenius rule increasing

temperature reduced viscosity and specific viscosity is reduced due to the freeness of solution.

From Viscometric analysis The viscosity of the larger scale formation of complexation were measured at 100 rpm was lower than the experimental-scale (13.5 ml starch solution) but not significant difference ( $P > 0.05$ ) with an approximately  $1,785.15 \pm 13.18$  and  $1,790.50 \pm 12.21$  cP, respectively. The lower the viscosity, the less was tendency for any further spontaneous reduction with surfactant mixture.

From the Tables 2 to 4 we are trying to identify the differences among the ionic and non-ionic surfactants in presence of polymeric starch the results show different between the respective two types surfactants. Not only this Triton-X-100 and Tween-20 are the non ionic but SDS and CTAB are ionic. Comparable study Ionic surfactants showed excellent results as reduced viscosities value and specific viscosities values. Here, we have been found that the increasing temperature the values of reduced viscosity and specific viscosity has decreased due to freeness of bonding in the complexes as a inclusion compound in between starch and ionic surfactants but in case of Triton-X-100 and Tween-20 no bond formation is formed due to the absence of dipole in that respective two non ionic surfactants (Fig.2(b)).

From the Fig.3 (a) it has been seen five distinct phases and they are 1L, S+L, LC, LC+S and S The one liquid phase is shown in the area ayb. This phase indicates 15% starch is solubilized in water / starch boundary line. The boundary line of CTAB / water indicates 25% of CTAB solubilized in this phase, the Solid + liquid phases are described by the area abcd. In the S+L phase water/CTAB boundary line, CTAB is 55 to 75% solubilized and starch is 25 to 65% solubilized in this phase. The most important and interesting liquid crystal phase (LC) is denoted by the region dcef. in the LC phase. 45 to 55% of CTAB is solubilized on the water/CTAB boundary line and also starch is 35 to 65% solubilized on the water/starch boundary line, LC+S phase: In this phase is shown in the area efgh. It is the big region and CTAB is 25 to 45% solubilized in the water/CTAB boundary line and starch is 65 to 95% solubilized on the water/starch boundary line and In the

corners of starch and CTAB the Solid phase is formed. The region of this phase is ghxz. S, The solid phase waists toward CTAB and starch corner but this area is most inactive where no interactions occur.

From the Fig.3 (b) it has been seen five distinct phases and they are 1L, S+L, LC, LC+S and S The one liquid phase (1L) has shown in the area ayb. This phase indicates 17% of starch is solubilized in starch/water boundary line. The boundary line of water / SDS shows that 25% of SDS has solubilized in the 1L phase. The solid + liquid phase (S+L) is denoted by abcd. The S+L phase formed is relatively small. The most important and interesting liquid crystal phase (LC) is denoted by the region cdef. In the LC phase 35 to 36% of starch is solubilized in the water /starch boundary line and less than 9 to 10% SDS is solubilized in this phase. The remaining big region denoted by efgh is the liquid crystal and solid phase. In this phase, water / SDS boundary line shows to 54 to 55% SDS and starch / water boundary line shows 65 to 95% starch is solubilized in this phase. The remaining region gxzh is the solid phase, S which is unreacted area. From the Fig.3(c) it has been seen three distinct phases and there are:- 1L, S+L and S. In the 1L phase 7.5% starch is solubilized. Above the 1L phase a large region ydac of S+L phase is formed. In the S+L phase the maximum of starch is 75%. In the region azc is the small area where only solid phase, S is found. The phase area is minimum. Though Tween-20 is liquid sustenance, its interaction is different from other ionic (CTAB, SDS) surfactant with natural polymeric starch. In this ternary phase there is no crystalline phase due the non ionic surfactant of Tween-20. In this phase there is no change in the region ydx, here any type of chemical reaction did not occur due to non-ionic surfactant of Tween-20. From Fig.(c) it have been seen that as a non ionic surfactant of Tween-20 any chemical reactions, H-bond formations, any type of crystallinity has not seen in Fig.3(c) not form which support Tween -20 have no effect on starch like polymer for better interactions. Adding starch on surfactant, the reaction mechanism can be explained here due to the complex formation in between amylose and ionic surfactant through the H- bond formation. Here, it is mentioned that from SEM

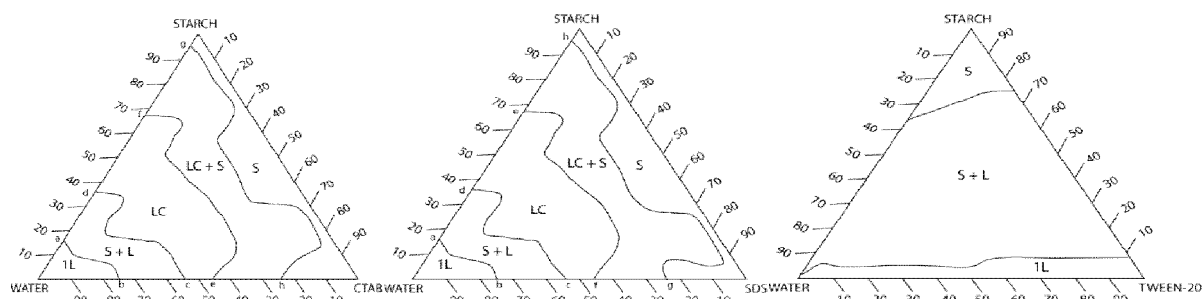


Fig. 3(a) : Starch+ Cationic CTAB+H<sub>2</sub>O (b) Starch+ Anionic SDS+H<sub>2</sub>O (c) Starch+Non-Ionic Tween-20+H<sub>2</sub>O

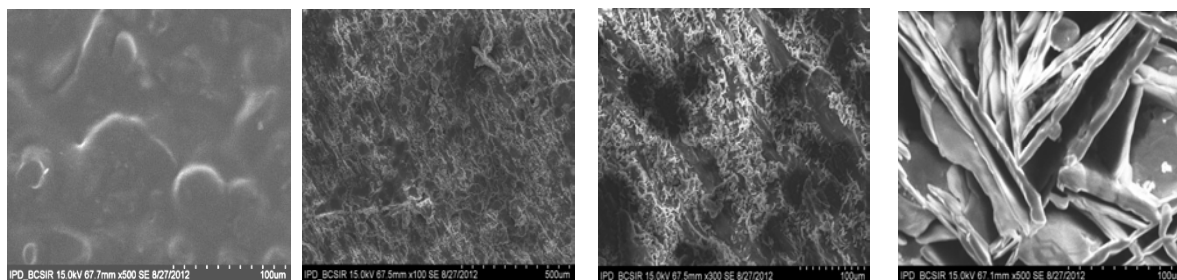


Fig. 4(a) : SEM Image of Starch (b) SEM Image of SDS (c) SEM Image of CTAB (d) SEM Image of CTAB+ Starch

various type of rupture are found on the surface area of complexes compared to other homogeneous surfaces obtained from SEM images of both individual samples.

From SEM Fig.4 (a), Fig.4(b) and Fig.4(c) have been found varieties types smooth, homogeneous surfaces but it is cleared to identify that in reacted starch-ionic surfactant complexes found in the Fig.4(d) has a lot of rupture region in the magnification area. Here, it has been identified smooth surfaces in only starch or only surfactant SEM image where as reacted complex film images showed a remarkable identity that have definite interaction may occur. So it is clearly decided that obviously interactions may occurred in starch and ionic surfactants complexes.

Amorphous and crystalline sections were examined from the X-ray diffractograms and the X-ray spectra are shown in Fig.5a. Peak baseline (white area) and smooth curve (bold area) were computer-plotted on the diffractogram which is shown in Fig.5b. The area above the smooth curve was the crystalline portion and the bold area above the peak baseline was the amorphous portion. The % crystallinity of complexes samples were calculated as the ratio of area of the crystalline sharp peak over the total area at angles between 10 and 90° using a computer program based on the methods of [29] Over all results discussions our observations are Starch/ surfactant interactions can be understood by assuming that the most important factors governing the behaviour on the systems are cooperative hydrophobic interactions between surfactant chains and

electrostatic interactions between cationic and anionic poly electrolytes Hydrophobic polymer /surfactant interactions are of minor importance. The enhanced surface activity of these systems at very low surfactant concentrations is due to formation of surface-active complexes by way of counter ion condensation.

Critical association concentrations are observed at concentrations well below the CMCs of the surfactants. There is a minimum hydrocarbon chain length of the surfactant and also a minimum degree of substitution of the starch to association between the starch and surfactant to take place. Associative phase separation occurs in extremely dilute systems when the charge ratio between the surfactants and the polymers is close to one. The effect of mixing on the micellization of the binary surfactant solutions can be described to a good approximation by taking into account only the effects of the volume difference between the hydrocarbon chains. Complex formation on starch with ionic surfactants depend on the chain-length difference in exactly the same way as for free mixed micelles. The separated complex phase is a hydrophobic, highly viscous and gel-like containing 40 to 60 w% of water. The high viscosity and low water content of the complex phase indicates that the interactions between the starch and ionic surfactants are very strong. The water content of the complex phase decreases when the chain length of the surfactant or the DS of the starch increases, indicating increased polymer/ surfactant interaction. With mixed surfactants the water content of

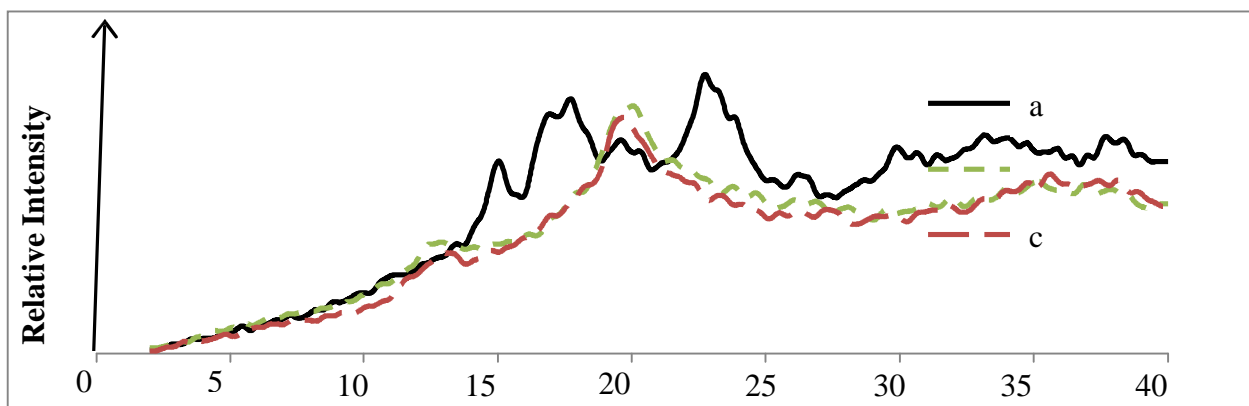


Fig. 5 : Calligraphy of X-ray diffraction patterns of **a**= black bold line normal potato starch, **b**= green dotted line Starch with CTAB and **c** = blue dotted line Starch with SDS



the complex phase increases when the chain length difference increases. The more surface-active component is strongly enriched in the polymer complexes. When excess surfactant is added, the separated complex phase redissolves completely or partly, depending on the charge density of the starch. XRD indicates that mixed micelles of alkanoates are prolate ellipsoids with the shorter chain surfactant enriched in the end-cups of the ellipsoid. XRD also indicates that in dilute solutions starch molecules are roughly cylindrical i.e. have helical conformation. When they associate with surfactants, aggregates are formed, in which a core of surfactant is surrounded by starch. The surfactant monomers do not form any micelle like aggregates in these complexes. The structure of the complexes resembles the inclusion complexes of amylose and surfactants. In mixed surfactant systems, the longer chain surfactant is enriched into the surfactant/starch complex. As the XRD results show, the charge equivalence starch/surfactant complexes consist of a lyotropic liquid crystalline phase. Depending on the chain length of the surfactant, temperature and concentration, the structure of the liquid crystalline phase is hexagonal, lamellar or cubic. The mesophases are the same as the pure surfactants in forming without any added polymer, but they form with starch at much lower surfactant concentrations. Thus, the polymer act as a huge and very effective counter ion that screens repulsion between the surfactant head groups and the surfactant aggregates. The gel-like starch/surfactant complex phases have high viscosity and elasticity. They show pseudoplastic behaviour. The rheological behaviour of the complexes is described by the Hershel-Bulkley model. The rheological data for the complex phases are compatible with the finding that the surfactants form liquid crystalline structures with the polymer anchored to the surfactant aggregates.

#### IV. CONCLUSIONS

Starch/surfactant interactions can be understood by assuming that the most important factors governing the behaviour on the systems are cooperative hydrophobic interactions between surfactant chains and electrostatic interactions between cationic and anionic polar part of Hydrophobic polymer /surfactant interactions are of minor importance. The rheological data for the complex phases are compatible with the finding that the surfactants form liquid crystalline structures with the polymer anchored to the surfactant aggregates by H- bond formation. In this study, the interactions between surfactants and starch have been investigated within different contexts. Systematic investigations such as: surface tension, viscosity and ternary phase on the effect of the surfactant structure (head group and chain length) on the temperature-induced changes of potato starch granules in excess of

water which reducing environmental hazards protecting our global green chemistry.

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## Ni-NP/Ni Anodes with Varying Ni: NP Ratio Prepared by Electrodeposition for the Direct Methanol Fuel Cell

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**Abstract-** Ni-NP/Ni anodes with varying Ni:NP ratio were prepared by electrodeposition of a thin Ni-NP (Nickel-Natural Phosphate) catalyst layer onto Ni plate for a direct methanol fuel cell (DMFC). The morphology and structure of the catalyst layers were analyzed by TEM and EDX. The catalyst coating layer shows an alloy character. The results show that these electrodes are very active for the methanol oxidation and that the optimum Ni: NP surface coverage was 50: 50 at % for DMFC operating at room temperature.

*GJRE-C Classification : FOR Code: 850301, 090499*



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Sadiki Lamari Abdelmajid <sup>α</sup>, Salah Eddine El Qouatli <sup>σ</sup> & Abdelilah Chtaini <sup>ρ</sup>

**Abstract-** Ni-NP/Ni anodes with varying Ni:NP ratio were prepared by electrodeposition of a thin Ni-NP (Nickel-Natural Phosphate) catalyst layer onto Ni plate for a direct methanol fuel cell (DMFC). The morphology and structure of the catalyst layers were analyzed by TEM and EDX. The catalyst coating layer shows an alloy character. The results show that these electrodes are very active for the methanol oxidation and that the optimum Ni: NP surface coverage was 50: 50 at % for DMFC operating at room temperature.

## I. INTRODUCTION

Direct methanol fuel cell (DMFC) is a new generation alternative energy harnessing device [1-3]. The electrochemical processes that yield energy are essentially pollution free. Water formed during the operation of the device is beneficial in space travel and submarines. Applications of fuel cells are diverse ranging from stationary (individual homes or district schemes) or mobile (transformation as cars, buses, etc.), mobile phones and lap top computers [4, 5].

The liquid feed direct methanol fuel cell (DMFC) is considered as a potential power source for stationary and transportation application because of characteristics such as simple construction, easy operation, liquid fuel and high efficiency [5, 6]. However, obstacles still prevent their widespread commercial applications [7, 8], e.g. low activity of methanol electrooxidation catalysis, methanol crossover from the anode to the cathode, carbon dioxide gas management and water management [9].

Hydrogen is currently the only practical fuel for use in the present generation of fuel cells. The main reason for this is its high electrochemical reactivity compared with that of the more common fuels from which it is derived, such as hydrocarbons, alcohols, or coal.

Also, its reaction mechanisms are now rather well understood [10, 11] and are characterized by the relative simplicity of its reaction steps, which lead to no side products.

Pure hydrogen is attractive as a fuel, because of its high theoretical energy density, its innocuous combustion product (water), and its unlimited availability so long as a suitable source of energy is available to decompose water. One of the disadvantages of pure hydrogen is that it is a low density gas under normal conditions, so that storage is difficult and requires considerable excess weight compared with liquid fuels.

Methanol has been considered for fuel cell power generation for a number of years because it can be processed into a hydrogen-rich fuel gas fairly, easily and efficiently, by steam or auto thermal reforming. Methanol, as a liquid fuel is easily transported and stored in comparison to hydrogen gas. The methanol fuel has a superior specific energy density (6000 Wh/kg) in comparison with the best rechargeable battery, lithium polymer and lithium ion polymer (600 Wh/kg) systems. This means longer conservation times using mobile phones, longer times for use of laptop computers and more power available on these devices to support consumer demand. Another significant advantage of the direct methanol fuel cells over the rechargeable battery is their potential for instantaneous refueling [12-13]. The methanol fuel cells in alkaline solutions have many advantages such as increasing their efficiency, a wider selection of possible electrode materials, a better efficiency of oxygen cathode, and the oxidation reactions of organic fuels exhibit almost no sensitivity to the surface structure [14, 15].

Moreover, the use of the methanol fuel cells is limited by the poor anode performance and counteract the poisoning effects at the cathode due to the methanol cross-over [16, 17]. In the electro oxidation of methanol, the electrode material is clearly an important parameter where a high efficient electro catalyst is needed.

The use of Pt alone is limited by the adsorption of several incompletely oxidized intermediates [18, 19] which provoke deactivation of platinum surface. For this reason, there are several studies investigating modified Pt, for methanol electro oxidation, such, Pt-Ru [20], Pt-Pb [21], Pt-Ni and Pt-Ru-Ni [22].

Several studies of the alcohols electro oxidation on Ni have been investigated [23-26] due to its surface oxidation properties.

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In this work, Ni-NP/Ni anodes with varying Ni: NP ratio are prepared by electrodeposition NiNP catalyst layer onto Ni plate and tested for methanol oxidation.

## II. EXPERIMENTAL DETAILS

### a) Electrode Preparation

Electrochemical measurements were performed on nickel plate electrode of apparent surface area of 1 cm x cm. Catalyst coating on the nickel plate was prepared using electrodeposition. The nickel electrode was mechanically polished using smooth paper, and then it was subsequently degreased with acetone and rinsed with distilled water. Before each experiment, the electrode surface was activated via the cyclic voltammetry at scan rate of 500 mV/s for 20 cycles. The working electrode (Ni and NP-Ni) were sealed in Teflon jacket. The apparent surface area was calculated from geometrical area and the current density was referred to it.

### b) Reagents

A natural phosphate (NP) used in this work was obtained in the Khouribga region (Morocco) [27]. Prior to use, this material was treated by techniques involving attrition, sifting, calcinations (900 °C), washing, and recalcination [28]. Measurements were carried out in aerated 1M KOH at room temperature (20 ± 0.2 °C).

### c) Instrument

Cyclic voltammetry, chronoamperometry, chronopotentiometry, impedance spectroscopy (EIS), polarization curves and square wave voltammetry were carried out with a Voltalab potentiostat (Model PGSTAT 100, Echochemie B.V., Utrecht, the Netherlands) driven by the general purpose electrochemical systems data processing software (Voltalab Master 4 software). The electrochemical cell was configured to work with three electrodes using prepared electrode as the working electrode, platinum plate (1cm x cm) for counter electrode and Ag-AgCl as reference electrode.

## III. RESULTS AND DISCUSSION

### a) Natural Phosphate Characteristics

The surface structure of natural phosphate (NP) was observed using scanning electron microscopy (Fig. 1). The treatment of NP describes above lead to a fraction between 100 µm and 400 µm that is rich in phosphate and as can be seen that compact natural phosphate appearance was evident. The treated NP has following chemical composition:

CaO (54.12%), P<sub>2</sub>O<sub>5</sub> (34.24%), F<sup>-</sup> (3.37%), SiO<sub>2</sub> (2.42%), SO<sub>3</sub> (2.21%), CO<sub>2</sub> (1.13%), Na<sub>2</sub>O (0.92%), MgO (0.68%), Fe<sub>2</sub>O<sub>3</sub> (0.36%), K<sub>2</sub>O (0.04%) and several metals in the range of ppm.

It is seen that the morphology of NP catalyst surface is porous.

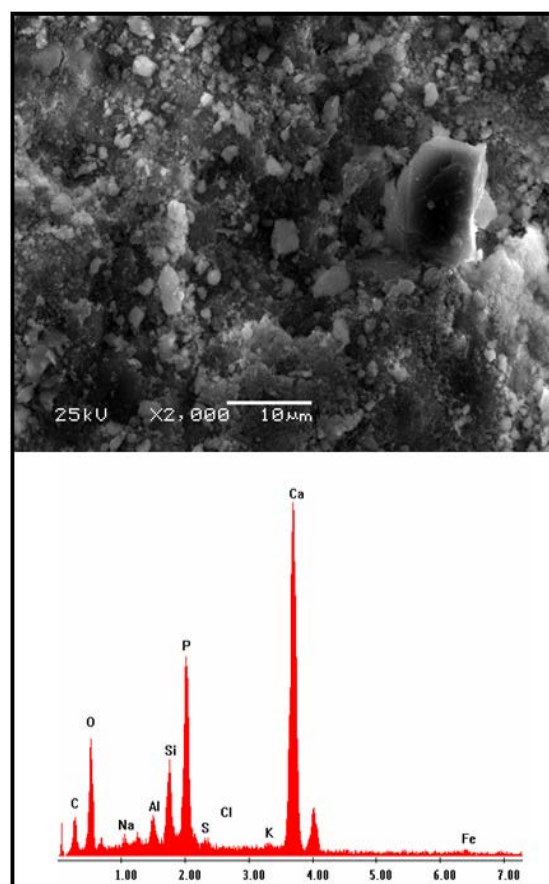


Figure 1 : Scanning Electron Micrograph of Natural Phosphate

The crystal-structure of matter is similar to that of fluorapatite (Ca<sub>10</sub> (PO<sub>4</sub>)<sub>6</sub>F<sub>2</sub>), as shown by X-ray diffraction (Fig. 2) and infrared emission spectroscopy (Fig. 3). The network of fluorapatite is very tolerant of substitutions in vacant sites, such as Ca can be replaced by Sr, Pb, Co and Na, PO<sub>4</sub> by AsO<sub>4</sub>, VO<sub>4</sub> and SO<sub>4</sub> and F<sup>-</sup> can be replaced by OH<sup>-</sup> and Cl<sup>-</sup>. The phosphate has a low specific surface area of about 1m<sup>2</sup> g<sup>-1</sup>.

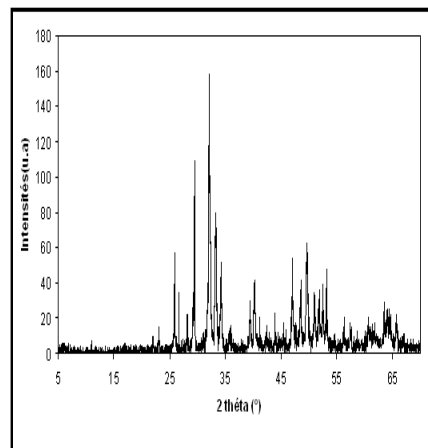


Figure 2 : XRD Pattern of the Natural Phosphate



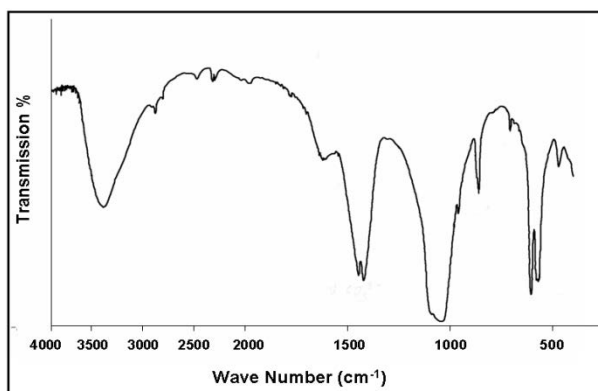


Figure 3 : IR Spectra of the Natural Phosphate

b) Electrochemical Characterization of Prepared Electrode

The cyclic voltammograms (CVs) of the Ni plate, NP/Ni plate and NP-Ni/Ni electrodes were recorded in the supporting electrolyte (1M KOH solution). The change of the CVs shapes (Figs. 4, 5 and 6), before and after modification, is a major sign that Ni electrode was, effectively, modified with NP and NP-Ni layers.

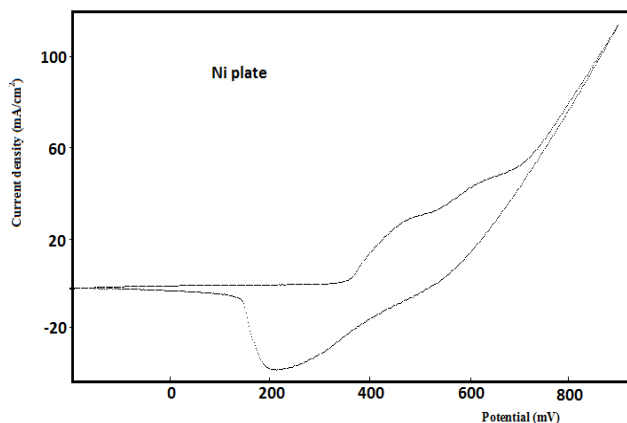


Figure 4 : Cyclic Voltammogram Obtained by Ni Electrode, at 100 Mv/S, In 1M KOH Solution

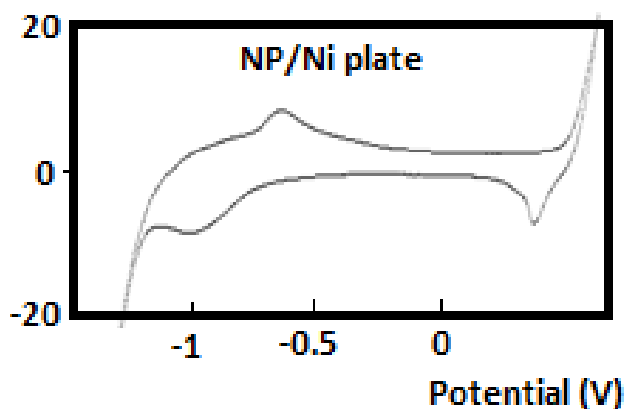


Figure 5 : Cyclic Voltammogram Obtained by NP/Ni Electrode, at 100 Mv/S, In 1M KOH Solution

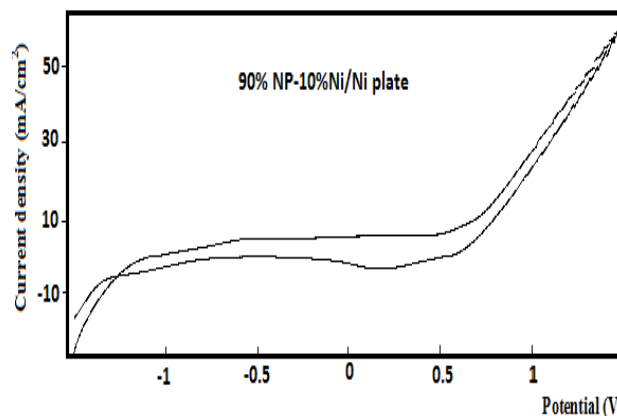


Figure 6 : Cyclic Voltammogram Obtained by NP-Ni/Ni Electrode, at 100 Mv/S, In 1M KOH Solution

The collected parameters deduced from the polarization curves, recorded respectively, for the prepared electrodes, such as the corrosion potential ( $E_{corr}$ ), corrosion current density ( $J_{corr}$ ), cathodic and anodic Tafel slopes ( $\beta_c$  and  $\beta_a$ ), are shown in Table 1.

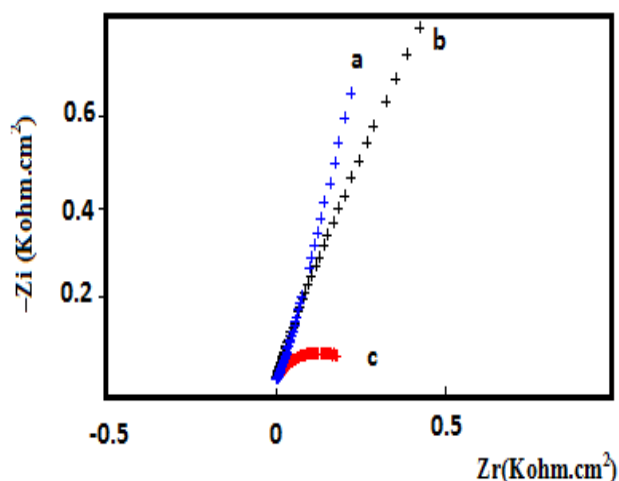
We find that the Ni electrode is more resistant to corrosion than Np/Ni electrode modified. However the addition of a small amount of Ni to NP matrix results in improved surface.

Table 1 : Electrochemical Parameters

Electrodes	$E(i=0)$ (mV)	$R_p$ (ohm/cm²)	$I_{corr}$ (mA/cm²)	$B_a$ (mV)	$B_c$ (mV)
Ni	232.6	279.01	0.0669	88.4	-250.7
NP/Ni	-1098.7	58.21	1.026	560.1	-216.8
NP90%-Ni 10% / Ni	-970.6	124.66	0.4852	440.1	-247.7

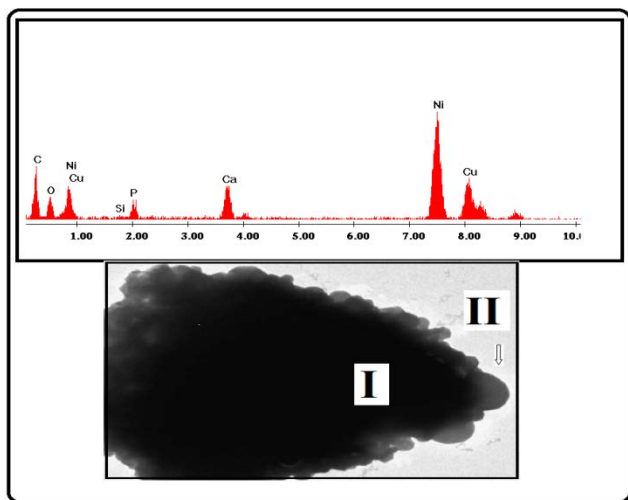
The results of the potentiodynamic polarization experiments were confirmed by impedance spectroscopy measurements. The Nyquist plots for the tested electrodes (Ni, NP/Ni and Ni-NP/Ni) in alkaline solution are presented in Fig. 7.

The locus of Nyquist plots is regarded as one part semi circle in NP/Ni electrode (curve c), but in Ni and Ni-NP/Ni electrodes (curves a and b), the plot was not perfect semi circle. This feature had been attributed to frequency dispersion of interfacial impedance.



**Figure 7 :** Electrochemical Impedance Spectroscopy Recorded for Ni, NP/Ni and NP-Ni/Ni Electrodes, in 1M KOH Solution

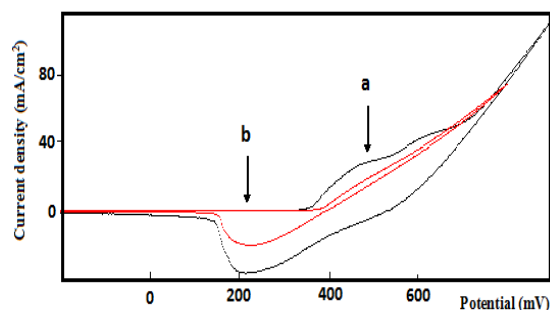
A typical EDX spectrum is shown in Fig. 8 the atomic ratio of Ni (electrodeposited onto NP/Ni) in Ni NP/Ni electrode is about (40%). The characteristic peak of Ni can not be found in Fig. 8. It indicates that Ni plate is almost fully covered by the Ni-NP catalyst layer.



**Figure 8 :** EDX Spectra Analysis of the Ni-NP/Ni (40 At % Ni) Scanning Electron Micrographs of the Cross-Section of Ni-NP/Ni Anode: I: Ni And II: Ni-NP Catalyst

#### c) Methanol Oxidation

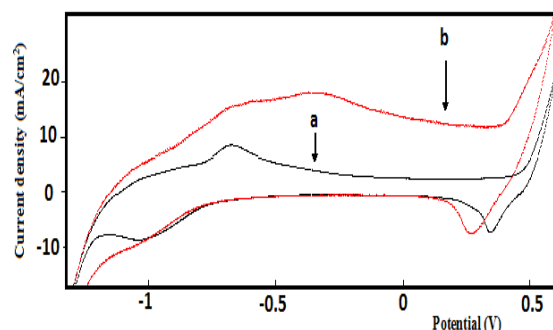
Fig.9 compares the CVs of the Ni plate electrode recorded in alkaline solution containing methanol (curve b) and without methanol (curve a). It is clearly seen that nickel has little activity of methanol oxidation.



**Figure 9 :** Cvs Recorder for Ni Plate Electrode, in N<sub>2</sub>-Saturated 1M KOH A- in Absence of Methanol, B- in Presence of 0.1M Methanol

The cyclic voltammetry (Cv) for the NP/Ni electrode in KOH is shown in fig. 10. The CV was carried out to analyze the activity of the synthesized catalyst towards methanol electro-oxidation in alkaline media. It should be recorded here that the using of natural phosphate (NP), electrodeposited onto Ni plate, to catalyze the methanol oxidation has proved better performance than using pure nickel.

It could be noticed from fig. 10 that the onset potential for methanol electro-oxidation of in alkaline medium using the catalyst NP/Ni was about -1000 mV. But the current densities are very low.



**Figure 10 :** Cvs Recorder for NP/Ni Plate Electrode, in N<sub>2</sub>-Saturated 1M KOH A- in Absence of Methanol, B- in Presence of 0.1M Methanol

Using Ni-NP/Ni (atomic ratio Ni: NP=1:9) catalyst the onset potential for electro-oxidation of methanol in alkaline medium was 400 mV, with higher current densities (fig. 11). The change of onset potentials could be associated to the structure change after Ni was alloyed with NP. The current density values have been increased in case of Ni-NP/Ni, more than NP/Ni and Ni plate, due to the increase in active electrochemical surface area.

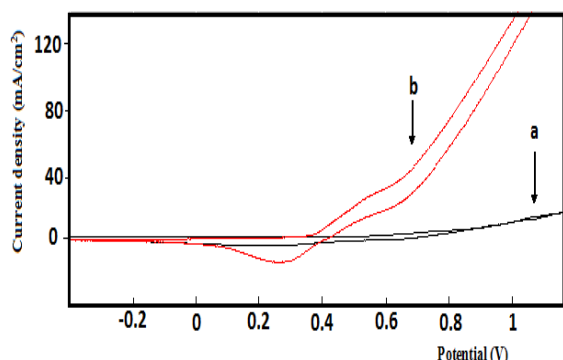


Figure 11 : Cvs Recorder for Ni-NP/Ni Plate Electrode, in N<sub>2</sub>-Saturated 1M KOH A- In Absence of Methanol, B- In Presence of 0.1M Methanol

It is found from figs. 12 and 13 that the current density and the power density increase with increasing methanol concentration. However, both the power density and the current density become stable at about 0.5 mol/L of methanol, probably due to saturation of the catalyst surface Ni-NP/Ni.

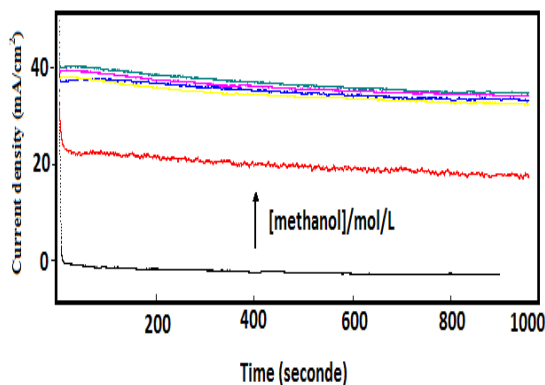


Figure 12 : Transient Current Density of Methanol Electro-Oxidation, at Ni-NP/Ni Electrode, at Room Temperature, in Alkaline Solution Containing Different Concentrations of Methanol

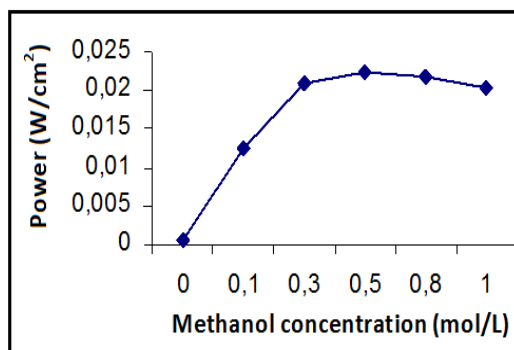


Figure 13 : Influence of Methanol Concentration on Power Density

## IV. CONCLUSION

A novel electrode was fabricated and tested for methanol electro-oxidation. The experimental results indicate that the Ni-NP/Ni catalyst has proved better activity, and the power density of the cell increases with methanol concentrations, and the maximum power density of 25 mW/cm<sup>2</sup> was achieved with 0.3 M methanol concentration.

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## Adsorption Kinetics of Sulfates by Anion Exchange Resin Containing Pristine Multiwalled Carbon Nano Tubes

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**Abstract-** Since the documented discovery of carbon nanotubes (CNT) in 1991 by Iijima and the realization of their unique physical properties, including mechanical, thermal, and electrical, many investigators have endeavored to fabricate advanced CNT composite materials that exhibit one or more of these properties. The sulfates removal from synthetic water by High cross-linked polystyrene divinyl benzene resin containing pristine multiwalled carbon nano tubes was studied at batch experiments in this study. The effect of pH, contact time, sulfates concentration and adsorbent dose on the sulfates sequestration was investigated. The optimum conditions were studied on Saline water as a case study. The results showed that with increasing of the absorbent amount; contact time and pH improve the efficiency of sulfates removal. The maximum sulfates uptake was obtained in pH and contact time 3.0 and 120 min, respectively. Also, with increasing initial concentration of sulfates in water, the efficiency of sulfates removal decreased. The obtained results in this study were matched with Freundlich isotherm and pseudo second order kinetic. The maximum adsorption capacity ( $X_m$  or  $K$ ) and constant rate were found 0.024 (mg/g) and 2.083 (mg/g.min), respectively. This study also showed that in the optimum conditions, the sulfates removal efficiency from Saline water by 0.1 mg/L sulfates was 85.64%. Eventually, High cross-linked polystyrene divinyl benzene resin containing pristine multiwalled carbon nano tubes is recommended as a suitable and low cost absorbent to sulfates removal from aqueous solutions.

**Keywords:** adsorption, Freundlich isotherm, Langmuir isotherm, water, hard, nano.

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# Adsorption Kinetics of Sulfates by Anion Exchange Resin Containing Pristine Multiwalled Carbon Nano Tubes

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**Abstract-** Since the documented discovery of carbon nanotubes (CNT) in 1991 by Iijima and the realization of their unique physical properties, including mechanical, thermal, and electrical, many investigators have endeavored to fabricate advanced CNT composite materials that exhibit one or more of these properties. The sulfates removal from synthetic water by High cross-linked polystyrene divinyl benzene resin containing pristine multiwalled carbon nano tubes was studied at batch experiments in this study. The effect of pH, contact time, sulfates concentration and adsorbent dose on the sulfates sequestration was investigated. The optimum conditions were studied on Saline water as a case study. The results showed that with increasing of the adsorbent amount; contact time and pH improve the efficiency of sulfates removal. The maximum sulfates uptake was obtained in pH and contact time 3.0 and 120 min, respectively. Also, with increasing initial concentration of sulfates in water, the efficiency of sulfates removal decreased. The obtained results in this study were matched with Freundlich isotherm and pseudo second order kinetic. The maximum adsorption capacity ( $X_m$  or  $K$ ) and constant rate were found 0.024 (mg/g) and 2.083 (mg/g.min), respectively. This study also showed that in the optimum conditions, the sulfates removal efficiency from Saline water by 0.1 mg/L sulfates was 85.64%. Eventually, High cross-linked polystyrene divinyl benzene resin containing pristine multiwalled carbon nano tubes is recommended as a suitable and low cost adsorbent to sulfates removal from aqueous solutions.

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## I. INTRODUCTION

Carbon nanofibers and nanotubes are promising to revolutionise several fields in material science and are a major component of nanotechnology. Further market development will depend on material availability at reasonable prices. Nanotubes have a wide range of unexplored potential applications in various technological areas such as aerospace, energy, automobile, medicine, or chemical industry, in which they can be used as gas adsorbents, templates, actuators, composite reinforcements, catalyst supports,

probes, chemical sensors, nano pipes, nano-reactors etc. Sulfates is widely popular for this application because of its ability to kill bacteria and other disease-causing organisms at relatively low concentrations. Carbon nano tubes (CNTs) have superior material properties such as high chemical stability, aspect ratio, mechanical strength and activated surface area as well as outstanding electrical properties, which make them good electro active material candidates for super capacitors. The electrodes made from CNTs exhibit a unique pore structure for charge storage; however, there are limitations for further increasing the effective surface area of the CNTs, as well as relatively high materials cost which limit the commercial application of CNTs based super capacitors. To improve the performance of CNTs, they are composited with conductive polymers and metal oxides. This section will summarize the recent development of CNTs based nano composites for super capacitor applications. Techniques that can be used to synthesize CNTs include Arc discharge, chemical vapour deposition, and laser ablation. Synthesized single-walled CNTs by dc arc discharge of a graphite rod under helium gas using Ni, Co, and FeS as catalysts. Then they prepared single-walled CNTs-polypyrrole (PPY) nano composite using in situ chemical polymerization of pyrrole monomer in solution with single-walled CNTs suspension. As-grown single-walled CNTs, pure PPY, and single-walled CNT-PPY nano composite powder can form by the in situ chemical polymerization. The as-grown single-walled CNTs are randomly entangled and cross-linked, and some carbon nano particles are also observed. The sulfate binds and destroys the outer surface of bacteria and viruses thereby preventing waterborne diseases. The sulfate that is not used is called free residual sulfates. There are many health concerns regarding residual sulfates in water. This is mainly because sulfates is not naturally needed by the body and as a halogen, it tends to inactivate enzymes [1]. Chlorinated water contains chemical compounds called tri haloethanes which are carcinogens. Bathing with chlorinated water could have the same effect as drinking because of dermal absorption of sulfates [2]. Other concerns are aesthetic such as sulfates taste and odour. These concerns inform the need for an effective means of

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water de chlorination after disinfection at low cost. In Europe, the most common means of destroying microorganisms in water system is the use of ozone treatment. In Nigeria, sulfates is extensively used in water treatment because it is cheaper than ozone. High cross-linked polystyrene divinyl benzene resin are good at removing sulfates residual from water but the actual rates of sulfates removal by High cross-linked polystyrene divinyl benzene resin obtained from different local raw materials need to be determined. Fast adsorption rate and high adsorption capacity are important and must be considered in the selection of an activated carbon for a given purpose. The performance of High cross-linked polystyrene divinyl benzene resin is indicated by its adsorptive characteristics, which is derived from the specific surface area, pore size and pore volume of the High cross-linked polystyrene divinyl benzene resin. In this study, adsorption isotherms and sulfates removal kinetics from water using High cross-linked polystyrene divinyl benzene resin from selected local raw materials were considered [3].

## II. MATERIALS AND METHODS

### a) Instruments and Materials

Styrene (Merck), Benzoyl peroxide were supplied (Merck, as cross-linking agent), and used without further purification, Dichloromethane, Acetone, Trimethylamine (Fluka, assay 45% in water, as amination agent), Toluene, from Egyptian market and were used as received. Pristine Multi-wall carbon nano tubes (MWCNTs) were synthesized in Nano lab inside EPRI and its purity is 95 wt. %, average diameter of 15 nm and lengths ranging from 0.5  $\mu\text{m}$  to 1  $\mu\text{m}$ . Surfactant, Hydroxyl propyl cellulose (HB) (Aquator co. with MS= 3, density= 0.5mg/l, MP=450-500  $^{\circ}\text{C}$ ). Paraformaldehyde (Merck), 1,4-butandiol (Merck), zinc chloride (Merck, as catalyst for chloromethylation step), hydrochloric acid (Merck), nitric acid (Merck), methanol (Merck), sodium chloride, sodium hydroxide, magnesium chloride, calcium chloride (ADWIC).

### b) Synthesis of MWCNTs (Chemical Vapour Deposition)

The catalytic chemical vapor deposition (CCVD) process for synthesis of multi-walled carbon nanotubes (MWCNTs) was carried out at atmospheric pressure using horizontal fixed bed flow reactor. In this respect 0.5 g of the catalyst powder (Co-MgO) [4] was dispersed onto a quartz tube of 4 cm length and placed in the center of a silica tube with a diameter of 3 cm and 100 cm length fitted within a furnace. The temperature was increased to 700  $^{\circ}\text{C}$  under a mixture of hydrogen (50 sccm) and nitrogen (150 sccm) and kept at this temperature for 1 h to deoxidize the catalyst. Subsequently, the hydrogen gas was changed to 50 sccm of natural gas diluted with 150 sccm nitrogen for a

certain reaction time of 4 h. Finally, the system was cooled down to ambient temperature under nitrogen flow of 150 sccm [5, 6].

### c) Synthesis of PSDVB and PS-DVB-CNT beads

2 g polymer of PS seeds was placed in a 500mL three neck flask. Then, the emulsified solution containing 30mL SDS aqueous solution (0.2%, w/v) was added and stirred overnight. Afterwards, another emulsified mixture consisted of 30 g organic compounds and 250 mL HB aqueous solutions (1%, w/v) were prepared by an Ultrasonic Compact Hielscher UP200Ht (200W, 26 kHz) with a nominal frequency and power of 28 kHz and 600 W, and poured into the flask for swelling. Hence, the organic compounds used is consisted of styrene, divinylbenzene, MWCNTs, toluene, BPO and SDS, where, the amount of toluene equaled to the sum of styrene and divinylbenzene, while BPO is 1.5% of the sum (w/w), and the amount of SDS is 0.25% of the HB aqueous solution (w/v), finally, the amount of MWCNTs is 1% (w/w) was attempted to prepare PSDVB-CNT particles. After 24 hours the temperature was increased to 70  $^{\circ}\text{C}$  under nitrogen atmosphere and lasted another 24 hours. Subsequently, the resulting beads were washed successively with hot water and alcohol. Then the beads were extracted with toluene for 48 hours. After washing and drying, the resulting particle size in the stationary phase was about 50-150  $\mu\text{m}$  [7, 8].

### d) Chloromethylation of Copolymer Resins

A 2.0176 g of resins, 2, 5 ml of chloromethyl methyl ether, 1.1530 g of  $\text{ZnCl}_2$  and 10 ml of nitrobenzene were added to a 50 ml flask equipped with refluxing condenser and anhydrous  $\text{CaCl}_2$  tube. After stirring at room temperature for 5–10 min, the chloromethylation reaction was carried out at 45 $^{\circ}\text{C}$  for 4 hours. After that, 0.5770 g of  $\text{ZnCl}_2$  was added to the system, the polymerization was carried out at 80 $^{\circ}\text{C}$  for 12 hours. The resulting polymer washed with hot deionized water, and extracted with acetone in a Soxhlet apparatus. The product was dried under vacuum to give 2.3907 g of resin [6].

### e) Amination of Chloromethylated Copolymer

A 250 mL round bottom flask fitted with magnetic stirrer was charged with 5 g of polymer beads, 150 ml of acetonitrile and 30 ml of tri ethylamine, then the mixture was purged with nitrogen, deaerated under vacuum and the entire setup was placed in an oil bath over a magnetic stirrer and the mixture was refluxed at 70  $^{\circ}\text{C}$  for 3 days with a continuous pumping of nitrogen into the reaction flask. The quaternized beads were filtered and successively washed with acetonitrile and methanol followed by drying under vacuum at 60  $^{\circ}\text{C}$  [7].

### f) Effect of pH value on sulfates adsorption

The effect of pH on sulfate ions adsorption was carried out using initial chloride ion concentration 100 mg/L onto 0.1 g/L of resins at different pH values (1-9).

The solution was agitated for 120 min and the residual sulfate ion in solutions was determined, also the distribution of chloride ion depends upon pH of the solution were studied [8].

#### g) Effect of sorbent dose

The effects of changing initial resin dosage in the sorption medium on removal of sulfates ions in terms of adsorption capacity was examined at different values including 0.10, 0.050, 0.1, and 0.2, 0.3, 0.35 g resin/L and equilibrated for 24 hours, while other operational parameters such as temperature and initial chlorine and sulfates ions concentration of solutions were kept constant ( $T=30^{\circ}\text{C}$ ,  $C_0=100\text{ mg M/L}$ ,  $\text{pH}=\text{natural}$ ) [9].

#### h) Effect of initial sulfates concentration

1000 mg/L stock solution of sulfate ions was diluted to obtain standard solutions containing 10–100 mg/L of chloride ion. A 100 mL of chloride ion solutions of a desired concentration was adjusted to a desired pH, then poured in 300 mL reaction bottles and known amounts of ion exchange resins were added. The solution pH was adjusted by using buffer solutions. The solutions were agitated at 150 rpm for a predetermined period at  $30^{\circ}\text{C}$  in a shaking incubator (JEIO TECH SI-900R). The resins were separated and the filtrate was analyzed by ICP for chloride ion content [9].

### III. RESULTS AND DISCUSSION

#### a) Effect of pH value on sulfates uptake

The pH of the ion exchange system determines the adsorption capacity due to its influence on the surface properties of the different ionic forms of the sulfate solutions. Changes of the adsorption capacity of sulfate onto prepared resins with pH are shown in Figure (1). It was observed that the maximum removal percentage (85%) occurred at pH 2.5. Maximum adsorption at acidic pH indicates that the low pH leads to an increase in  $\text{H}^+$  ions on the resins surface which results in significantly strong electrostatic attraction between positively charged PS-DVB-P-MWCNTs resins surface and sulfate ions. The removal percentage of sulfate adsorption gradually decreased to 18 at pH 10. Adsorption of sulfate onto ion exchange resins were not significant at pH values greater than 6.0 due to anions competition to be exchanged on the surface of the prepared ion exchange resins of which  $\text{OH}^-$  predominates. The pH of exchanged ions is a very important character that determines the pH at which the ion exchange resins surface has net electrical neutrality. At this value, the acidic or basic functional groups no longer contribute to the pH of the solution [10].

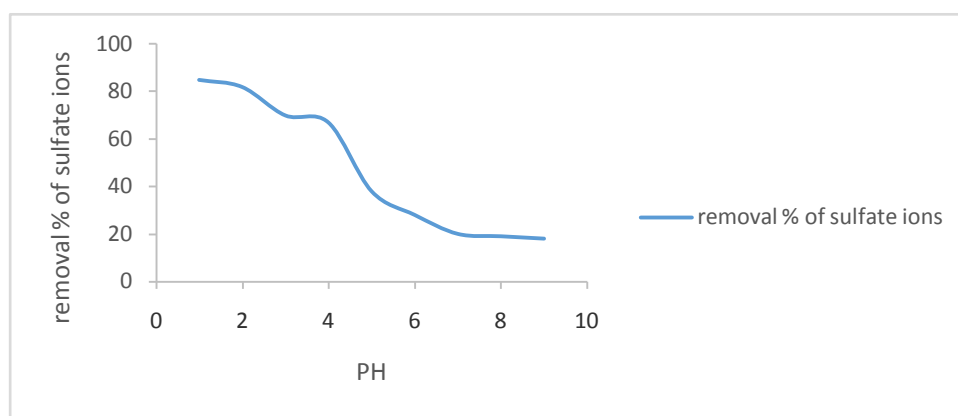


Figure 1 : Effect of pH value on sulfates uptake

#### b) Effect of initial sulfates concentration [11]

The amount of sulfate anions adsorbed for different initial concentrations onto anionic PS-DVB containing pristine multiwalled carbon nano tubes, resins is shown in Figures (2). The results show that the adsorption process is clearly time dependent. The amount of sulfate adsorbed (mg/g), increased with increase sulfate concentration and remained nearly constant after equilibrium time. It was shown that the adsorption at different initial concentration was rapid in the initial stages and gradually decreased with the progress of adsorption until the equilibrium reached 120 min. At low concentrations the ratio of available surface to the initial sulfate concentration is large, so the

removal becomes independent on initial concentrations. However, in the case of the high concentrations, this ratio is low, and the exchange rate then depends upon the initial concentration. In addition the curves are continuous leading to saturation, suggesting the monolayer coverage of sulfate on the surface of the adsorbent [11]. After a balance time of 120 minute, the adsorption capacity records an increase from 11 to 83.2 mg/g, for concentrations from 25 to 100 mg/L in PS-DVB resin containing pristine multiwalled carbon nano tubes. This may be due to the fact that at a chosen adsorbent dose, the number of active adsorption sites remains unchanged while with higher adsorbate concentrations, the adsorbate ions to be accommodate increase. At the

higher initial concentrations, the higher corresponding cumulative removal (mg/g) is dominated. The small amount of exchange sulfate ions evidence that the presence of carbon nano tubes create specialized

resins rate that satisfactory for chlorine ion more than sulfate ions and this reduces the competition between them which is clear in the resins containing MWCNTs.

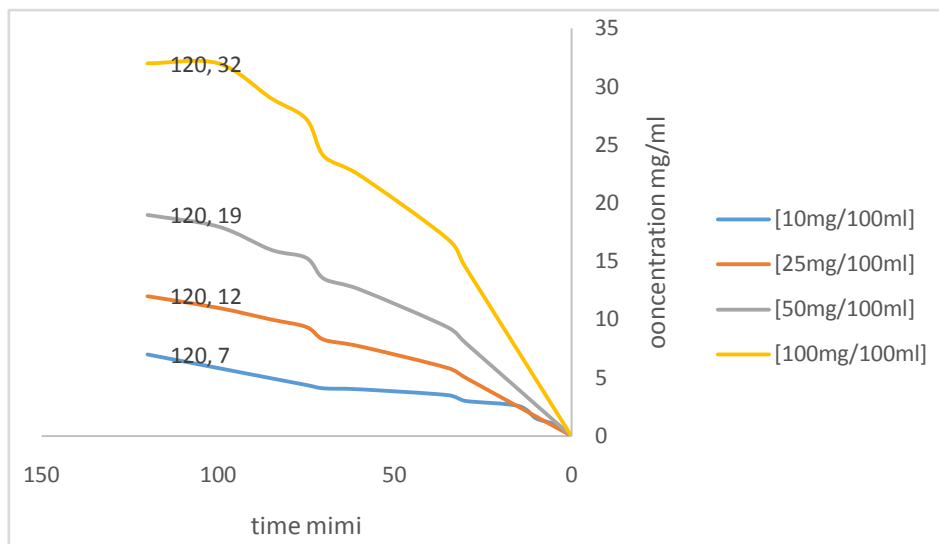


Figure 2 : Effect of initial sulfates concentration

#### c) Effect of sorbent dose

In order to examine the effect of the resins dosage on the removal efficiency sulfate, adsorption experiments were set up with various amounts of PS-DVB-P-MWCNTs resins (0.05-35 g/100mL) at initial sulfate concentration of 100 mg/100mL and at pH = 3.5. The effect of resins dosages on the amount of adsorbed sulfate has been shown in Figure (3). It was shown that the quantity of sulfate adsorbed increased in a significant values in the pondered range examined. In addition, we observe that the maximum of retention is obtained for a mass of 250mg of PS-DVB-P-MWCNTs

per 100mL of solution. Beyond an adsorbent dose of 250mg/100mL, the recovery % becomes constant. This is probably because the resistance to mass transfer of sulfate from bulk liquid to the surface of the adsorbent and the saturation of active sites, which becomes important at high adsorbent loading in the system in which the experiment was conducted. It might have happened that the higher dose causes particles aggregates and interference or repulsive forces between binding sites, therefore decreases the interaction of sulfate ions with the sorbent and reduces the total surface area of the adsorbent [11]

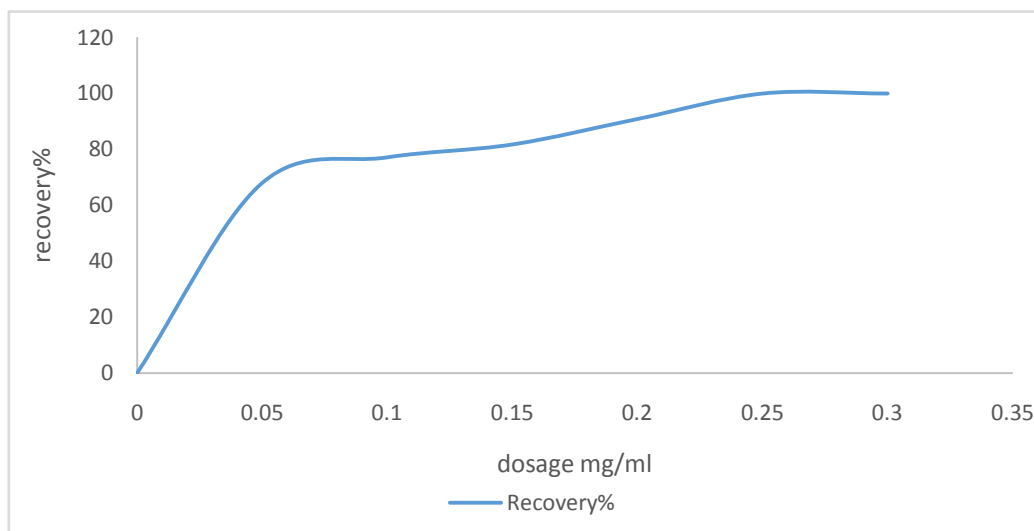


Figure 3 : Effect of sorbent dose

d) Isotherm data analysis

In this study, Freundlich, Langmuir isotherms were studied. The Table 1 gives the isotherms equation as well as constants. According to the results, the Freundlich isotherm was best fitted to represent the

equilibrium adsorption data than other isotherms (Figure 4,5). RL (separation factor) is the important factor in Langmuir isotherm that is the dimensionless constant[11].

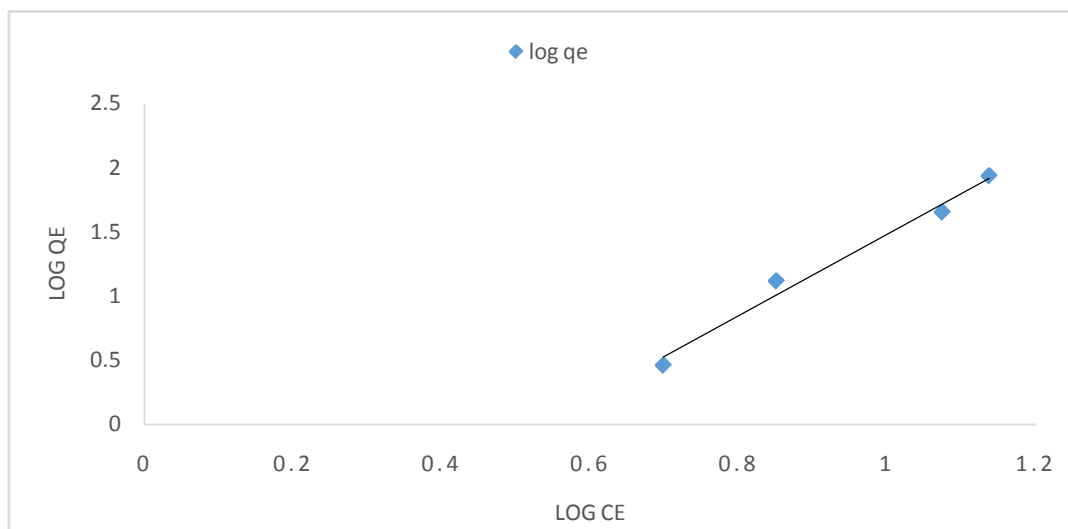


Figure 4 : Langmuir Isotherm data analysis

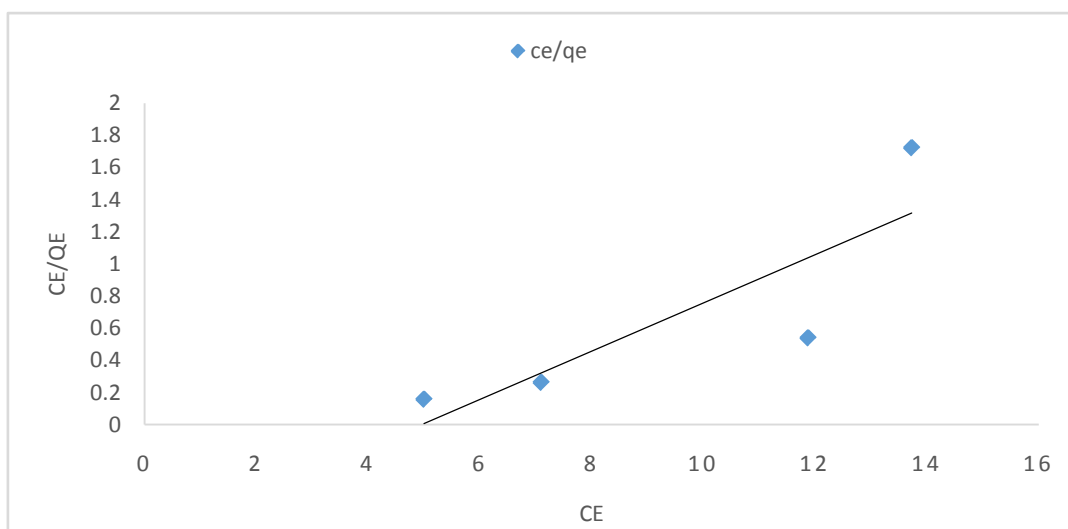


Figure 5 : Freundlich Isotherm data analysis

Table 1 : Summary of isotherm parameters for the adsorption of sulfate ions on anionic exchange resins

resins	Freundlich				Langmuir			
	1/n	n	K	R <sup>2</sup>	1/X <sub>m</sub>	X <sub>m</sub>	R <sup>2</sup>	K <sub>L</sub>
PS-DVB-P-MWCNTs	0.48	0.5873	2.083	38.66	0.8873	0.024	0.6269	41.66

e) Kinetic Studies

Sulfates removal from aqueous solution by resins may be presented by first-order (second-order, and Intraparticle diffusion kinetic models. Table 2 presents the kinetics equation and constant value for

sulfates adsorption on pumice. Sulfates-pumice interaction concurred showed good fit with pseudo-second order rate equation (R<sup>2</sup>=0.99). Figure 5 is shown pseudo-second order kinetic model [11].



Table 2 : Kinetic studies of sulfate ions sorption on PS-DVB-P-MWCNTs resin

Kinetic model	Nonmetals ions	Metal concentrations	Kinetic Parameter		
			R <sup>2</sup>	k <sub>L</sub> , ads or K <sub>d</sub>	q <sub>e</sub>
Pseudo first order model	SO <sub>4</sub> <sup>-2</sup>	10	0.9352	0.0036	0.9711
		25	0.9876	0.0024	1.386
		50	0.9798	0.0018	1.6875
		100	0.9622	0.0015	1.9874
Second order model	SO <sub>4</sub> <sup>-2</sup>	10	0.8532	0.0015	0.0955
		25	0.9954	0.0003	0.0401
		50	0.9909	0.0001	0.0203
		100	0.9739	4*10 <sup>-5</sup>	0.0102
Pseudo Second order model	SO <sub>4</sub> <sup>-2</sup>	10	0.8682	0.1233	5.7899
		25	0.9405	0.0374	5.3949
		50	0.9403	0.023	3.3543
		100	0.927	0.0142	1.8113
intraparticle diffusion model	SO <sub>4</sub> <sup>-2</sup>	10	0.9432	0.5903	0.2575
		25	0.9955	1.3191	2.3289
		50	0.995	2.1306	3.7614
		100	0.9886	3.6976	6.0978

#### IV. CONCLUSIONS

Removal of SO<sub>4</sub><sup>2-</sup> from aqueous solution onto anionic PS-DVB resin containing pristine multiwalled carbon nano tubes was carried out at room temperature. Results indicate that pH, initial sulfate concentration, stirring intensity, adsorbent dosage and foreign anions impacted sulfate specie removal: the SO<sub>4</sub><sup>2-</sup> uptake increased with the increase of initial sulfate concentration and decreased with increasing pH values. It is also seen that a further increase in adsorbent dose (greater than 0.1 mg/L) affects the uptake of sulfate adsorption greatly. The conditions of maximum adsorption of the sulfate anions were optimized. In nature and in normal treatments, the treated waters are usually at pH from 1 to 9, so the adsorption capacity of SO<sub>4</sub><sup>2-</sup> is about 2.25 mg/g at pH 3.5, for an adsorbent dosage of 0.1 mg/L, initial sulfate concentration of 50 mg/L, under a constant temperature of 25 ± 2 °C, and the equilibrium state was reached within 120 min of exposure time. The results showed that the adsorption of sulfate onto anionic PS-DVB resin containing pristine multiwalled carbon nano tubes followed pseudo-second-order rate kinetic predicting a chemisorption process. The results of present investigation show that the relatively low cost and high capabilities of the raw date palm seeds make them potentially attractive adsorbents for the removal of sulfate from aqueous solution. Further experiments need to be conducted to test the dynamic sorption of SO<sub>4</sub><sup>2-</sup> onto anionic PS-DVB resin containing pristine multiwalled carbon nano tubes in batch process.

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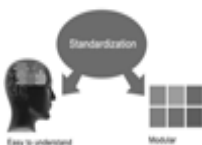
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**18. Pick a good study spot:** To do your research studies always try to pick a spot, which is quiet. Every spot is not for studies. Spot that suits you choose it and proceed further.

**19. Know what you know:** Always try to know, what you know by making objectives. Else, you will be confused and cannot achieve your target.

**20. Use good quality grammar:** Always use a good quality grammar and use words that will throw positive impact on evaluator. Use of good quality grammar does not mean to use tough words, that for each word the evaluator has to go through dictionary. Do not start sentence with a conjunction. Do not fragment sentences. Eliminate one-word sentences. Ignore passive voice. Do not ever use a big word when a diminutive one would suffice. Verbs have to be in agreement with their subjects. Prepositions are not expressions to finish sentences with. It is incorrect to ever divide an infinitive. Avoid clichés like the disease. Also, always shun irritating alliteration. Use language that is simple and straight forward. put together a neat summary.

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

**22. Never start in last minute:** Always start at right time and give enough time to research work. Leaving everything to the last minute will degrade your paper and spoil your work.

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

**24. Never copy others' work:** Never copy others' work and give it your name because if evaluator has seen it anywhere you will be in trouble.

**25. Take proper rest and food:** No matter how many hours you spend for your research activity, if you are not taking care of your health then all your efforts will be in vain. For a quality research, study is must, and this can be done by taking proper rest and food.

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



**27. Refresh your mind after intervals:** Try to give rest to your mind by listening to soft music or by sleeping in intervals. This will also improve your memory.

**28. Make colleagues:** Always try to make colleagues. No matter how sharper or intelligent you are, if you make colleagues you can have several ideas, which will be helpful for your research.

**29. Think technically:** Always think technically. If anything happens, then search its reasons, its benefits, and demerits.

**30. Think and then print:** When you will go to print your paper, notice that tables are not be split, headings are not detached from their descriptions, and page sequence is maintained.

**31. Adding unnecessary information:** Do not add unnecessary information, like, I have used MS Excel to draw graph. Do not add irrelevant and inappropriate material. These all will create superfluous. Foreign terminology and phrases are not apropos. One should NEVER take a broad view. Analogy in script is like feathers on a snake. Not at all use a large word when a very small one would be sufficient. Use words properly, regardless of how others use them. Remove quotations. Puns are for kids, not grunt readers. Amplification is a billion times of inferior quality than sarcasm.

**32. Never oversimplify everything:** To add material in your research paper, never go for oversimplification. This will definitely irritate the evaluator. Be more or less specific. Also too, by no means, ever use rhythmic redundancies. Contractions aren't essential and shouldn't be there used. Comparisons are as terrible as clichés. Give up ampersands and abbreviations, and so on. Remove commas, that are, not necessary. Parenthetical words however should be together with this in commas. Understatement is all the time the complete best way to put onward earth-shaking thoughts. Give a detailed literary review.

**33. Report concluded results:** Use concluded results. From raw data, filter the results and then conclude your studies based on measurements and observations taken. Significant figures and appropriate number of decimal places should be used. Parenthetical remarks are prohibitive. Proofread carefully at final stage. In the end give outline to your arguments. Spot out perspectives of further study of this subject. Justify your conclusion by at the bottom of them with sufficient justifications and examples.

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

## INFORMAL GUIDELINES OF RESEARCH PAPER WRITING

### Key points to remember:

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

### Final Points:

A purpose of organizing a research paper is to let people to interpret your effort selectively. The journal requires the following sections, submitted in the order listed, each section to start on a new page.

The introduction will be compiled from reference matter and will reflect the design processes or outline of basis that direct you to make study. As you will carry out the process of study, the method and process section will be constructed as like that. The result segment will show related statistics in nearly sequential order and will direct the reviewers next to the similar intellectual paths throughout the data that you took to carry out your study. The discussion section will provide understanding of the data and projections as to the implication of the results. The use of good quality references all through the paper will give the effort trustworthiness by representing an alertness of prior workings.



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

### **General style:**

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

To make a paper clear

- Adhere to recommended page limits

Mistakes to evade

- Insertion a title at the foot of a page with the subsequent text on the next page
- Separating a table/chart or figure - impound each figure/table to a single page
- Submitting a manuscript with pages out of sequence

In every sections of your document

- Use standard writing style including articles ("a", "the," etc.)
- Keep on paying attention on the research topic of the paper
- Use paragraphs to split each significant point (excluding for the abstract)
- Align the primary line of each section
- Present your points in sound order
- Use present tense to report well accepted
- Use past tense to describe specific results
- Shun familiar wording, don't address the reviewer directly, and don't use slang, slang language, or superlatives
- Shun use of extra pictures - include only those figures essential to presenting results

### **Title Page:**

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



### Abstract:

The summary should be two hundred words or less. It should briefly and clearly explain the key findings reported in the manuscript-- must have precise statistics. It should not have abnormal acronyms or abbreviations. It should be logical in itself. Shun citing references at this point.

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

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

- Reason of the study - theory, overall issue, purpose
- Fundamental goal
- To the point depiction of the research
- Consequences, including definite statistics - if the consequences are quantitative in nature, account quantitative data; results of any numerical analysis should be reported
- Significant conclusions or questions that track from the research(es)

### Approach:

- Single section, and succinct
- As a outline of job done, it is always written in past tense
- A conceptual should situate on its own, and not submit to any other part of the paper such as a form or table
- Center on shortening results - bound background information to a verdict or two, if completely necessary
- What you account in an conceptual must be regular with what you reported in the manuscript
- Exact spelling, clearness of sentences and phrases, and appropriate reporting of quantities (proper units, important statistics) are just as significant in an abstract as they are anywhere else

### Introduction:

The **Introduction** should "introduce" the manuscript. The reviewer should be presented with sufficient background information to be capable to comprehend and calculate the purpose of your study without having to submit to other works. The basis for the study should be offered. Give most important references but shun difficult to make a comprehensive appraisal of the topic. In the introduction, describe the problem visibly. If the problem is not acknowledged in a logical, reasonable way, the reviewer will have no attention in your result. Speak in common terms about techniques used to explain the problem, if needed, but do not present any particulars about the protocols here. Following approach can create a valuable beginning:

- Explain the value (significance) of the study
- Shield the model - why did you employ this particular system or method? What is its compensation? You strength remark on its appropriateness from a abstract point of vision as well as point out sensible reasons for using it.
- Present a justification. Status your particular theory (es) or aim(s), and describe the logic that led you to choose them.
- Very for a short time explain the tentative propose and how it skilled the declared objectives.

### Approach:

- Use past tense except for when referring to recognized facts. After all, the manuscript will be submitted after the entire job is done.
- Sort out your thoughts; manufacture one key point with every section. If you make the four points listed above, you will need a least of four paragraphs.



- Present surroundings information only as desirable in order hold up a situation. The reviewer does not desire to read the whole thing you know about a topic.
- Shape the theory/purpose specifically - do not take a broad view.
- As always, give awareness to spelling, simplicity and correctness of sentences and phrases.

#### **Procedures (Methods and Materials):**

This part is supposed to be the easiest to carve if you have good skills. A sound written Procedures segment allows a capable scientist to replacement your results. Present precise information about your supplies. The suppliers and clarity of reagents can be helpful bits of information. Present methods in sequential order but linked methodologies can be grouped as a segment. Be concise when relating the protocols. Attempt for the least amount of information that would permit another capable scientist to spare your outcome but be cautious that vital information is integrated. The use of subheadings is suggested and ought to be synchronized with the results section. When a technique is used that has been well described in another object, mention the specific item describing a way but draw the basic principle while stating the situation. The purpose is to text all particular resources and broad procedures, so that another person may use some or all of the methods in one more study or referee the scientific value of your work. It is not to be a step by step report of the whole thing you did, nor is a methods section a set of orders.

#### **Materials:**

- Explain materials individually only if the study is so complex that it saves liberty this way.
- Embrace particular materials, and any tools or provisions that are not frequently found in laboratories.
- Do not take in frequently found.
- If use of a definite type of tools.
- Materials may be reported in a part section or else they may be recognized along with your measures.

#### **Methods:**

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

#### **Approach:**

- It is embarrassed or not possible to use vigorous voice when documenting methods with no using first person, which would focus the reviewer's interest on the researcher rather than the job. As a result when script up the methods most authors use third person passive voice.
- Use standard style in this and in every other part of the paper - avoid familiar lists, and use full sentences.

#### **What to keep away from**

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

#### **Results:**

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

The page length of this segment is set by the sum and types of data to be reported. Carry on to be to the point, by means of statistics and tables, if suitable, to present consequences most efficiently. You must obviously differentiate material that would usually be incorporated in a study editorial from any unprocessed data or additional appendix matter that would not be available. In fact, such matter should not be submitted at all except requested by the instructor.





## Content

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

### What to stay away from

- Do not discuss or infer your outcome, report surroundings information, or try to explain anything.
- Not at all, take in raw data or intermediate calculations in a research manuscript.
- Do not present the similar data more than once.
- Manuscript should complement any figures or tables, not duplicate the identical information.
- Never confuse figures with tables - there is a difference.

### Approach

- As forever, use past tense when you submit to your results, and put the whole thing in a reasonable order.
- Put figures and tables, appropriately numbered, in order at the end of the report
- If you desire, you may place your figures and tables properly within the text of your results part.

### Figures and tables

- If you put figures and tables at the end of the details, make certain that they are visibly distinguished from any attach appendix materials, such as raw facts
- Despite of position, each figure must be numbered one after the other and complete with subtitle
- In spite of position, each table must be titled, numbered one after the other and complete with heading
- All figure and table must be adequately complete that it could situate on its own, divide from text

### Discussion:

The Discussion is expected the trickiest segment to write and describe. A lot of papers submitted for journal are discarded based on problems with the Discussion. There is no head of state for how long a argument should be. Position your understanding of the outcome visibly to lead the reviewer through your conclusions, and then finish the paper with a summing up of the implication of the study. The purpose here is to offer an understanding of your results and hold up for all of your conclusions, using facts from your research and generally accepted information, if suitable. The implication of result should be visibly described. Infer your data in the conversation in suitable depth. This means that when you clarify an observable fact you must explain mechanisms that may account for the observation. If your results vary from your prospect, make clear why that may have happened. If your results agree, then explain the theory that the proof supported. It is never suitable to just state that the data approved with prospect, and let it drop at that.

- Make a decision if each premise is supported, discarded, or if you cannot make a conclusion with assurance. Do not just dismiss a study or part of a study as "uncertain."
- Research papers are not acknowledged if the work is imperfect. Draw what conclusions you can based upon the results that you have, and take care of the study as a finished work
- You may propose future guidelines, such as how the experiment might be personalized to accomplish a new idea.
- Give details all of your remarks as much as possible, focus on mechanisms.
- Make a decision if the tentative design sufficiently addressed the theory, and whether or not it was correctly restricted.
- Try to present substitute explanations if sensible alternatives be present.
- One research will not counter an overall question, so maintain the large picture in mind, where do you go next? The best studies unlock new avenues of study. What questions remain?
- Recommendations for detailed papers will offer supplementary suggestions.

### Approach:

- When you refer to information, differentiate data generated by your own studies from available information
- Submit to work done by specific persons (including you) in past tense.
- Submit to generally acknowledged facts and main beliefs in present tense.



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



# INDEX

---

---

## A

Alochrom · 13  
Anodized · 12  
Anomalous · 1  
Arrhenius · 19

---

## C

Compensates · 4  
Concentricity · 18  
Crevices · 13  
Cumulonimbus · 1

---

## D

Dirigible · 1, 7, 8  
Dissipating · 1

---

## E

Encircled · 1  
Epoxide · 11

---

## M

Megajoules · 3

---

## P

Permittivity · 24  
Preferably · 11, 16

---

## R

Rescuffing · 13

---

## S

Saturation · 4  
Stirred · 11, 13

---

## V

Vaporization · 5  
Violently · 1



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