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CHEMISTRY

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## CONTENTS OF THE ISSUE

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- i. Copyright Notice
- ii. Editorial Board Members
- iii. Chief Author and Dean
- iv. Contents of the Issue
  
1. Kinetics and Thermodynamics of  $\beta$  - 1 Endosulfan Pesticide Adsorption in Jordan Valley Soils. **1-10**
2. Studies on Alkyd Cross Linkable Acrylic Hybrid Emulsion for Coating Application. **11-19**
3. A Study on the Chemical Separation of *Ficus Rumphii* Blume Extract. **21-23**
4. Synthesis and Chemistry of Bis-imidazole Derivatives: A Review. **25-31**
  
- v. Fellows
- vi. Auxiliary Memberships
- vii. Preferred Author Guidelines
- viii. Index



# Kinetics and Thermodynamics of $\beta$ - 1 Endosulfan Pesticide Adsorption in Jordan Valley Soils

By Nader Mohmoud Sadeq

*Alexandria University*

**Abstract-** Knowledge of the  $\beta$ - endosulfan adsorption characteristics of soil is necessary for predicting its mobility and fate in soil environments. To examine the characteristics of  $\beta$ -endosulfan retention, sorption batch experiments were performed on three Xeropsamments soils having different properties. The sorption data were applied to various kinetics models. The results reveal that the tested models fitted the adsorption data in the order: first-order~modified Freundlich > parabolic diffusion > Elovich. The energy of activation ( $E_a$ ) and enthalpy ( $\Delta H^\#$ ), entropy ( $\Delta S^\#$ ), and free energy of activation ( $\Delta G^\#$ ) related to  $\beta$ - endosulfan sorption were calculated using the Arrhenius equation. The activation energy ( $E_a$ ) values (18.57 - 38.18 kJ mol<sup>-1</sup>) and the positive  $\Delta H^\#$  values characterize  $\beta$ -endosulfan adsorption process onto the sorbents studied as physisorption with an endothermic nature. The negative values of  $\Delta S^\#$  (-23– -17 J mol<sup>-1</sup>) obtained in this study suggest that the  $\beta$ -endosulfan sorption process involves an associative mechanism.

**Keywords:** equilibrium isotherm models, kinetic models, associative mechanism, physisorption.

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**Abstract-** Knowledge of the  $\beta$ - endosulfan adsorption characteristics of soil is necessary for predicting its mobility and fate in soil environments. To examine the characteristics of  $\beta$ - endosulfan retention, sorption batch experiments were performed on three Xeropsamments soils having different properties. The sorption data were applied to various kinetics models. The results reveal that the tested models fitted the adsorption data in the order: first-order~modified Freundlich > parabolic diffusion > Elovich. The energy of activation ( $E_a$ ) and enthalpy ( $\Delta H^\#$ ), entropy ( $\Delta S^\#$ ), and free energy of activation ( $\Delta G^\#$ ) related to  $\beta$ - endosulfan sorption were calculated using the Arrhenius equation. The activation energy ( $E_a$ ) values (18.57 - 38.18 kJ mol<sup>-1</sup>) and the positive  $\Delta H^\#$  values characterize  $\beta$ -endosulfan adsorption process onto the sorbents studied as physisorption with an endothermic nature. The negative values of  $\Delta S^\#$  (-23- -17 J mol<sup>-1</sup>) obtained in this study suggest that the  $\beta$ -endosulfan sorption process involves an associative mechanism.

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## I. INTRODUCTION

The increasing use of pesticides in the modern agricultural practices leads to several environmental problems in soil and water. Transport of pesticides sorbed onto soil particles can represent a significant pathway from the original land application to receiving water bodies (Sen and Khilar 2006). Identifying and understanding the mechanisms controlling the fate of pesticides in the soil environment are vital for both maintaining environmental quality and optimizing sustainable agriculture practices (Soulas and Lagacherie 2001). Endosulfan(6,7,8,9,10-hexachloro-1,5,5a,6,9,9 a-hexahydro-6, 9-methano-2,3,4-enzodioxathiepin-3-oxide)is a broad spectrum contact insecticide and acaricide that is used throughout the world on a wide variety of fruits, vegetables, cereals, and cotton, and ornamental plants in commercial agricultural settings. Endosulfan is classified in the U.S. Environmental Protection Agency toxicity category I. Technical grade endosulfan is composed of two stereo chemical isomers: "  $\alpha$  -endosulfan and  $\beta$ -endosulfan, in concentrations of approximately 70% and 30%, respectively (Herrmann 2002). The  $\alpha$ -isomer is more

volatile and dissipative, while the  $\beta$ -isomer is generally more adsorptive and persistent (Rice et al.1997). The moderately adsorptive and persistency properties of  $\beta$ - isomer enable it to stay in the environment for an extended period (US EPA2007). Endosulfan has been detected in the atmosphere, soils, sediments, estuaries, surface, ground and rain waters, and food stuffs (Spark and Swift 2002). Once endosulfan is applied to crops, it can either persist in soil as a sorbed phase or be removed through several physical, chemical, and biological processes (Siddique et al. 2003). Because elevated endosulfan concentrations could result in both acute and chronic risks of concern for terrestrial and aquatic organisms, knowledge of the endosulfan adsorption characteristics of soil is necessary for predicting their mobility and fate in soil environments. Extensive use of endosulfan more than a decade to control insects in Jordan Valley, east of west bank is of great concern regarding the potential transport in the environment. The use of uncontrolled and large amount of endosulfan may deteriorate the soil and groundwater quality. The fate of endosulfan as an insecticide has not been examined yet thoroughly in Palestine and specifically in the Jordan Valley which is considered the main vegetable production area in the country. The main objective of this study was to determine sorption kinetics and thermodynamics from batch experiments for  $\beta$ -endosulfan and to ascertain sorption characteristics in representative soils of Jordan valley. Such information is necessary for understanding the mechanisms involved in the adsorption of endosulfan and the prediction of its movement in Jordan valley soils in order to establish risk assessment and remediation strategies for endosulfan contaminated soils (Kumar and Philip 2006; Krishna and Philip2008).

## II. MATERIALS AND METHODS

### a) Study area

Soil samples were taken from three locations intensely cultivated with vegetable and extended from northern part to southern part of the Jordan valley. These locations represent Jordan valley soils and included Bardaleh in northern part of Jordan Valley, Al-Zubeidate in middle part of Jordan valley, and Al-Nwueimeh in southern part of Jordan Valley. The study

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area has 5654 ha of irrigable land and is located between 31° 46 – 32° 24 North latitudes and 35° 27 – 35° 34 East longitudes. The Jordan valley has semi-arid climate with almost no precipitation between April and September. The long-term mean annual precipitation is about 166 mm, the mean annual atmospheric temperature is about 29.6 C° and the evaporation is 1500 mm. The soils of the Jordan valley are mainly sandy loamy, slightly alkaline (pH 7.2- 7.8) and classified as Xeropsamments according to the USDA system. The farmers in these locations usually apply large quantities of endosulfan (Thionex® 35) for insect and pests control. The study area contains more than 125 wells, distributed from northern part of Jordan valley to southern part of Jordan valley mostly used for irrigation only 12 wells are used for drinking.

#### b) Soil samples

Three different soils were chosen to represent the most common types in Jordan valley, Bardaleh in northern part of Jordan valley, Al-Zubeidate in middle of Jordan Valley and Al-Nwueimeh in southern part of Jordan valley. 101 Three surface samples (0 – 30 cm) were collected from each location, air-dried, ground, sieved (< 2 mm). and preserved in air tight polyethylene bags before use. Selected physical and chemical properties of these samples were determined (Table 1).

#### c) Adsorption kinetic study

Kinetic retention using the batch method was used to quantify adsorption isotherms for  $\beta$ -endosulfan by the three studied soils at three different temperatures 298, 308 and 318K. Triplicate 2-g samples of each soil were placed in Teflon centrifuge tubes and mixed with 40-mL solution of 15 and 30 110 mg L<sup>-1</sup> initial  $\beta$ -endosulfan concentrations. The mixtures were

#### e) Kinetics Modeling

Four kinetic models were applied to the sorption data:

First-order rate [Elkhatib et al.2013]

Elovich (Elkhatib et al.1992)

Parabolic Diffusion (Elkhatib and Hern 1988)

Power Function (Elkhatib and Hern 1988)

Where

$q$  or  $q_t$  =  $\beta$ -endosulfan adsorbed (mg kg<sup>-1</sup>) at time  $t$ ,  $q_o$  =  $\beta$ -endosulfan adsorbed (mg kg<sup>-1</sup>) at equilibrium,  $k_a$  = apparent sorption rate coefficient,  $\alpha$  = the initial adsorption rate (mg g<sup>-1</sup> min<sup>-1</sup>),  $\beta$  = a constant related to the extent of surface coverage (mg g<sup>-1</sup>) ,  $a$  = a constant;  $k_d$  = apparent diffusion rate coefficient,  $q$  = adsorbed  $\beta$ -endosulfan (mg kg<sup>-1</sup>),  $C_o$ = initial  $\beta$ -endosulfan concentration (mg l<sup>-1</sup>),  $t$  = reaction time (min),  $k_a$ = sorption rate coefficient (min<sup>-1</sup>), and  $1/m$  = constant.

#### f) Statistical analysis

The linear least-squares optimization program provided by SAS PROC LIN (SAS Institute 2000) was utilized to obtain best-fit parameters which provide the best description of the adsorption data.

continuously shaken on a temperature-controlled shaker at 120 rpm and then centrifuged at 5,000×g for 10 min prior to sampling. After 0.5, 1, 2, 4, 8 and 24 h hours of reaction time, a 5-mL aliquot was taken, extracted with n-hexane solution and filtered through anhydrous sodium sulphate to remove moisture content and analyzed using HPLC, model (Perkin Elmer, USA). Amounts of endosulfan sorbed by the soil matrix were determined by the difference between the concentrations of the supernatant and that of the initial solutions.

#### d) Analysis of $\beta$ -endosulfan

The extracted samples were analyzed using high-performance liquid chromatography model (Perkin Elmer Series 200). Analyses were performed in isocratic system using a 325 reverse phase C18 column (25 cm x 4.6 mm id). For separation of endosulfan, a 70:30 mixture of methanol and water was passed through the column at a flow rate of 0.7 ml/min and the wavelength of the UV/visible detector was fixed at 214 nm (Siddique et al., 2003). Column temperature was ambient and injection was made with a 50- $\mu$ l SGE syringe and a manual loop injector (20- $\mu$ l). Sample filtration was carried out 125 with solvent compatible Gellman syringe filter of 0.20- $\mu$ m GHP membrane. The eluent was degassed by purging with helium. Laboratory and instrumental blanks were frequently analyzed to ensure the absence of contaminants, or interference arising from samples or laboratory handling. Recoveries, calculated from spiked matrixes, were greater than 90%.Detection limits (LOD), ranged between 0.08 and 0.33 ng mL<sup>-1</sup> in agreement with values reported in the literature (Keith et al., 1983).

$$\ln(q_o - q) = a - k_a t \quad (1)$$

$$q_t = (1/\beta) \ln(\alpha \beta) + (1/\beta) \ln t \quad (2)$$

$$q = a + k_d t^{1/2} \quad (3)$$

$$q = k_a C_o t^{1/m} \quad (4)$$

### III. RESULTS AND DISCUSSION

#### a) $\beta$ -endosulfan Sorption vs. time

Sorption of  $\beta$ -endosulfan on the three studied soils versus time at two initial concentrations and a

temperature of 298K are illustrated in Fig.1. In the three studied soils the sorption of  $\beta$ -endosulfan kinetics exhibited an immediate rapid sorption by which about 73% -92% of initially  $\beta$ -endosulfan sorbed in the first hour followed by a slow sorption at 298K. The initial sorption rate for  $\beta$ -endosulfan was high as a large number of adsorption sites were available at the onset of the process. Adsorption slowed down in later stages because after some time the remaining vacant surface sites may be difficult to occupy due to the repulsive forces between  $\beta$ -endosulfan molecules on the solid and liquid phase (Anirudhan and Ramachandran 2007). When initial  $\beta$ -endosulfan concentrations were changed (15 and 30 mg/L) at temperature 298K; the amount sorbed per unit mass showed an increase with rise in initial  $\beta$ -endosulfan concentrations (Fig.1). This is due to the increase in the mass driving force which allows more  $\beta$ -endosulfan molecules to pass from the solution to the adsorbent surface. At low initial  $\beta$ -endosulfan concentration, the ratio of  $\beta$ - endosulfan in solution to the available sorption sites is small and consequently the sorption is dependent of the initial concentration, but as the concentration of the  $\beta$ -endosulfan increase, the competition for sorption sites becomes fierce (Sarvanane et al. 2002). Similar results were reported by Ismail et al. (2002) when studying adsorption of  $\beta$ -endosulfan on soils, they found that sorption of  $\beta$ -endosulfan increased with increasing solution concentrations.

#### b) Kinetics Modeling

The kinetics of  $\beta$ -endosulfan sorption on the three studied soils were analyzed using various kinetic models; first-order, Elovich, intraparticle diffusion model, and, modified Freundlich. The conformity between experimental data and the model predicted values was expressed by the coefficient of determination ( $R^2$ ) values and the standard error of estimate (SE). Based on  $R^2$  and SE values (Table2), the modified Freundlich and the first-order models best described  $\beta$ -endosulfan sorption on the three studied soils. The parabolic diffusion law described the adsorption data of  $\beta$ -endosulfan satisfactorily as the  $R^2$  values were quite high and SE values were low (Table 2). The empirical Elovich model did not describe the  $\beta$ -endosulfan reaction as the  $R^2$  values were lower and SE values were quite high for this model (Table 2). The order of application of various kinetic models to describe  $\beta$ -endosulfan adsorption data was first-order~modified freundlich>parabolic diffusion>Elovich Sorption processes in soil systems are complex and often involving some changes taking place during it, e.g. (1) An increase in vacant site numbers as sorption progress act. (2) Changes in solution conditions such as ionic strength. Therefore, careful selection of appropriate rate models capable of describing the inherently complex and system-specific dynamics of sorption processes is necessary for

accurate fate and transport modelling (Elkhatib et al.1992; Xie et al. 2011;193 Saha et al. 2013).

#### c) Effect of initial concentrations and temperatures on $\beta$ -endosulfan sorption

First-order equation was among the best kinetic equations studied 195 to describe the rate of  $\beta$ -endosulfan sorption as evidenced by the overall high values of  $R^2$  and low SE values (Table 2). Therefore, the first order rate equation was employed (Table3 and Fig.2) to analyze the sorption rate of  $\beta$ -endosulfan onto the soils studied. To ascertain that a reaction is a first order, a change in only one parameter (e.g., initial concentration) should generate parallel kinetic plots resulting from similar apparent rate coefficients. Fig.3 shows the first order kinetic plots for  $\beta$ -endosulfan sorption at initial concentrations of 15 and 30 mg L<sup>-1</sup> on Al-Zubeidate soils at 298K. The slopes (apparent rate coefficients) correspond well and are nearly equal confirming that the reactions are first order. For example, at 15 and 30 mg  $\beta$ -endosulfan L<sup>-1</sup> concentrations,  $\beta$ -endosulfan sorption on Al-Zubeidate soil at 298 K resulted in apparent rate coefficients of 0.0032 and 0.0035 respectively (Table 3).

Effect of temperature on  $\beta$ -endosulfan sorption was studied in all the three soils in the range of 298–318K at 15 and 30 mg L<sup>-1</sup> initial  $\beta$ -endosulfan concentrations. The  $\beta$ -endosulfan sorption rate on the studied soils, from greatest to least, at all three temperatures was as follows: Al-Zubeidate, Al-Nwueimeh and Bardaleh soils (Table 3). As the temperature of the reaction increased from 298 to 318K, with all other reaction conditions remaining constant, the  $\beta$ -endosulfan sorption increased on all soil sorbents. Elshafei et al. (2009) observed that the adsorption rate of cadusafos on soil at 298, 308 and 318K increased with increasing temperature. This work shows that at a low temperature (298K),  $\beta$ -endosulfan sorption is relatively slow, compared with sorption observed at 318K in the laboratory. The enhanced adsorption with temperature may be attributed to either increase in the number of active binding sites available for adsorption on the adsorbent surface or due to the decrease in the boundary layer thickness surrounding the adsorbent, and thereby decreased 219 boundary layer mass transfer resistance owing to the decrease in the viscosity of the solution. Often soil temperatures can rise above 318K in arid soils, indicating that sorption rates in the field can be even faster than reported here and thus allow higher soil surface loading levels.

#### d) Activation Energy and Thermodynamic Parameters of $\beta$ -endosulfan adsorption

By applying first order kinetic model and plotting  $\ln k_a$  vs.  $1/T$  for each soil concentration studied (Fig.4), where "T" is the temperature in Kelvin and " $k_a$ " is the apparent sorption rate coefficient, the following linear relationship is obtained:



$$\ln k_a = (-E_a/RT) + \ln A$$

Where

R= gas constant (8.314 kJ/mol),

A= universal frequency factor.

It can be seen that the slope is given by  $-E_a/R$  and the intercept by  $\ln A$ . Therefore, the thermodynamic parameters can be obtained using the following equations:

$$\Delta H^\# = E_a - RT$$

$$(\Delta S^\# / R) = \ln A - 1/n \text{ (BTe/h)}$$

$$\Delta G^\# = \Delta H^\# - T\Delta S^\#$$

Where

$\Delta H^\#$  = enthalpy of activation

$\Delta S^\#$  238 = entropy of activation

$\Delta G^\#$  = free energy of activation

B = Boltzman's constant

h = Planck's constant

e = electronic charge

The values of  $E_a$  for  $\beta$ -endosulfan sorption on the studied soils followed the range 21.25 to 22.47, 18.57 to 20.67 and 38.07 to 38.18 kJ mol<sup>-1</sup> for Al-Nnuweimeh, Al-Zubeidate and Bardaleh soils, respectively (Table 4). The energy of activation measures the magnitude of the forces to be overcome during the process of sorption reactions and may give an idea about the type of sorption. The activation energy for physical adsorption is usually less than 42 kJ mol<sup>-1</sup>, and higher values represent chemical reaction process as chemical adsorption is specific and involves forces much stronger than in physical adsorption (Elkhatib et al. 1993; Sparks 2003). In the present study, the values of activation energy confirm that the nature of  $\beta$ -endosulfan sorption onto studied soil is physical adsorption. The finding is in accordance with the observations of previous studies by Gulen et al. (2005). They found that, the value of activation energy ( $E_a$ ) for azinphosmethyl sorption onto pyrolyzed Horse shoe sea crab shell was about 1.52 kJ mol<sup>-1</sup>, this value is within the range of physical adsorption.

In order to get an insight whether the sorption process follows an activated complex, it is necessary to consider the thermodynamic activation parameters of the process. Values of enthalpy of activation ( $\Delta H^\#$ ), entropy of activation ( $\Delta S^\#$ ), and 258 free energy of activation ( $\Delta G^\#$ ) were computed and presented in Table 4. The values of enthalpy of activation ( $\Delta H^\#$ ) followed the range 18.77-19.99, 16.10-18.19 and 35. 60- 36.82 KJ mol<sup>-1</sup>, for Al- Nuweimeh, Al-Zubeidate and Bardaleh soils, respectively. Data in Table 4 showed that enthalpy of activation values decrease with increasing initial concentration signifying less energy requirements for the reaction system as a result of increasing initial  $\beta$ -endosulfan concentration in to the studied soils. The positive values of  $\Delta H^\#$  imply that the  $\beta$ -endosulfan adsorption process is endothermic and the magnitude of  $\Delta H^\#$  may indicate the type of binding mechanism

involved, i.e., physical and/or chemical sorption. The heat evolved during physical adsorption is in the range 2.1–< 40 kJ mol<sup>-1</sup>, while the heats of chemisorptions is about 40 kJ mol<sup>-1</sup>, a value that has been recognized in the literature as the transition boundary between both types of sorption processes (Liu and Liu, 2008). The calculated values of  $\Delta H^\#$  for  $\beta$ -endosulfan sorption were lower than 40 kJ mol<sup>-1</sup>, indicative of physical adsorption mechanism. This observation agrees with results reported by Memon et al. (2007) for the adsorption of methyl parathion onto chestnut shells. The values of  $\Delta G$  were in the range 87.59 to 87.79, 86.99 to 87.22, and 89.34 to 89.61 kJ mol<sup>-1</sup>, for Al- Nnuweimeh, Al-Zubeidate and Bardaleh soils, respectively (Table 4). The positive values of ( $\Delta G^\#$ ) indicate that sorption reactions require some energy from an external source to convert reactants into products and the nonspontaneous nature of  $\beta$ -endosulfan sorption onto the studied soil. The values of  $\Delta S^\#$  were in the range -23.08 to -22.74, -23.77 to -23.15 and -18.02 to -17.88 J mol<sup>-1</sup> K<sup>-1</sup>, for Al- Nuweimeh, Al-Zubeidate and Bardaleh soils, respectively (Table 4). The negative  $\Delta S^\#$  values (i.e., entropy decreases as a result of sorption) suggests the stability of sorption process with no structural change at solid-liquid interface. This occurs as a result of redistribution of energy between the sorbate and the sorbent. Before sorption occurs, the  $\beta$ -endosulfan molecules near the surface of the sorbent will be less ordered than in the subsequent sorbed state. As a result, the distribution of rotational and translational energy among a small number of molecules will decrease with increasing sorption by producing a negative value of  $S^\#$ . Additionally, the magnitude and sign of  $\Delta S^\#$  gives an indication whether the sorption reaction is an associative or dissociative mechanism.

Entropy values  $\geq 10$  J mol<sup>-1</sup> generally imply a dissociative mechanism (Atwood, 1997). Therefore, the negative values of  $\Delta S^\#$  (-23– -17 J mol<sup>-1</sup>) obtained in this study suggest that the  $\beta$ -endosulfan sorption process involves an associative mechanism. Similar results were reported by Yedla and Dikshit (2001) for endosulfan sorption onto wood charcoal.

#### IV. CONCLUSION

The results of the present study showed that The Al-Zubadate soil had the highest affinity for  $\beta$ -endosulfan sorption while the Bardaleh soil exhibited the least affinity;  $\beta$ -endosulfan sorption on Al-Nuweimeh soil was intermediate between that observed for Al-Zubadate and Bardaleh soils. Adsorption rate constant of  $\beta$ -endosulfan were highest for Al-Zubadate soil whereas, it was the least for Bardaleh soil, these suggest that the  $\beta$ -endosulfan have affinity to calcium carbonate and organic matter. Adsorption of  $\beta$ -endosulfan in all soils followed Freundlich isotherm model and it is inferred that the sorption was monolayer.

The influence of temperature on  $\beta$ -endosulfan sorption onto the soils were observed that an increase in the temperature for 298K to 318K led to an increase in percentage sorption for all studied soils the increase in sorption could be due to changes in pore size or an increase in kinetic energy of the sorption with increase in temperature suggests endothermic nature. The activation energy ( $E_a$ ) for sorption of  $\beta$ -endosulfan onto studied soils was determined using the Arrhenius equation, the values of activation energy for  $\beta$ -endosulfan sorption less than 42 kJ mol<sup>-1</sup>. Since  $E_a$  values < 42 KJ mol<sup>-1</sup> indicates that diffusion controlled process, it is concluded that  $\beta$ -endosulfan adsorption for the studied soils is a diffusion controlled reaction. The positive values of ( $\Delta H^\circ$ ) suggest that the sorption reactions are energy consuming process. The enthalpy ( $\Delta H^\circ$ ) of sorption reaction were positive suggest that sorption process were endothermic reaction. The small negative values for ( $\Delta S^\circ$ ) suggesting that  $\beta$ -endosulfan sorption for the studied soils is associative mechanism. The positive values of ( $\Delta G^\circ$ ) which are an indication of a nonspontaneous sorption process that is, due to the endothermic character. The magnitude of ( $\Delta G^\circ$ ) increased with the rise in temperature.

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**Table 1:** Selected physical and chemical characteristics of the three different soils used for the present study

| Characteristics                            | Al-Nwueimeh soil | Al-Zubeidate soil | Bardaleh soil |
|--|------------------|-------------------|---------------|
| pH #                                       | 7.76±0.2         | 7.28±0.007        | 7.64±0.05     |
| EC <sup>#</sup> (dsm <sup>-1</sup> )       | 2.25±0.02        | 21.41±0.01        | 3.49±0.007    |
| Total carbonate <sup>#</sup> (%)           | 39.14±0.3        | 35.74 ±0.4        | 12.43±0.3     |
| Sand (%)                                   | 75.55± 1.2       | 71.27 ± 1.3       | 69.61±0.98    |
| Silt (%)                                   | 10.4 ±1.5        | 11.38 ± 1.2       | 18.34±0.86    |
| Clay (%)                                   | 14.05± 1.9       | 17.35±1.5         | 12.05±1.3     |
| Texture <sup>§</sup>                       | sandy loam       | sandy loam        | sandy loam    |
| Organic matter <sup>¥</sup> (%)            | 1.37±0.04        | 3.25±0.06         | 1.04±0.04     |
| CEC <sup>§§</sup> cmol(+).kg <sup>-1</sup> | 13.755 ±0.4      | 35.815±0.3        | 14.651±0.2    |

# Soil: water ratio 1:2 ## Nelson,1982 § Day, 1965 § Rhoades ,1982 ¥ Nelson and Sommers,1982

**Table 2:** Range and mean values of coefficient of determination ( $R^2$ ) and standard error of estimates (SE) for different kinetics models fitted to the  $\beta$ -endosulfan sorption on the three studied soils

| Model                  | $R^2$        |       | SE          |       |
|------------------------|--------------|-------|-------------|-------|
|                        | Range        | Mean  | Range       | Mean  |
| <b>Kinetics Models</b> |              |       |             |       |
| Modified Freundlich    | 0.962-986    | 0.978 | 0.035-0.093 | 0.065 |
| First order            | 0.922***-988 | 0.948 | 0.001-0.13  | 0.075 |
| Elovich                | 0.882**-984  | 0.937 | 0.33-0.79   | 0.498 |
| Parabolic Diffusion    | 0.939-987    | 0.971 | 0.11-0.65   | 0.313 |

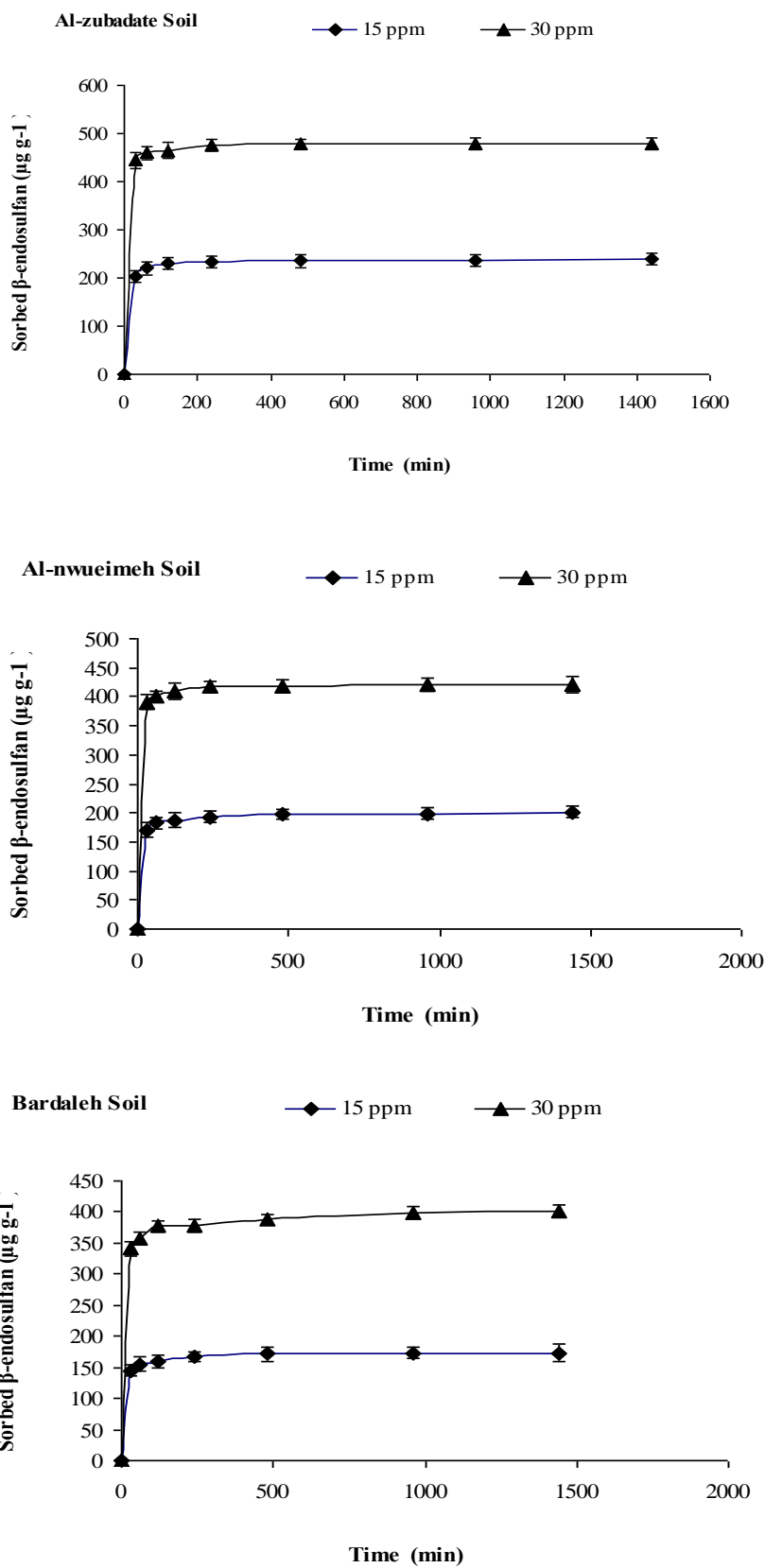
\*\*, \*\*\* significant at  $p < 0.01$  and  $p < 0.001$  respectively

**Table 3:** Calculated first order equation parameters  $\beta$ -endosulfan sorption for the three studied soils under isothermal conditions

| Initial Conc.<br>(mg L <sup>-1</sup> ) | Al-Nuweimeh soil                             |                         | Al-Zubeidate soil                            |                         | Bardaleh soil                                |                         |
|--|--|-------------------------|--|-------------------------|--|-------------------------|
|  | ka<br>min <sup>-1</sup><br>x 10 <sup>3</sup> | a<br>μg g <sup>-1</sup> | ka<br>min <sup>-1</sup><br>x 10 <sup>3</sup> | a<br>μg g <sup>-1</sup> | ka<br>min <sup>-1</sup><br>x 10 <sup>3</sup> | a<br>μg g <sup>-1</sup> |
| <b>298K</b>                            |  |                         |  |                         |  |                         |
| 15                                     | 2.61   | 2.28                    | 3.22   | 2.11                    | 1.21   | 2.47                    |
| 30                                     | 2.82   | 2.47                    | 3.54   | 2.33                    | 1.31   | 2.59                    |
| <b>308K</b>                            |  |                         |  |                         |  |                         |
| 15                                     | 3.42   | 1.28                    | 4.43   | 1.07                    | 2.22   | 1.12                    |
| 30                                     | 3.71   | 2.41                    | 4.71   | 2.25                    | 2.64   | 2.59                    |
| <b>318K</b>                            |  |                         |  |                         |  |                         |
| 15                                     | 4.63   | 1.26                    | 5.4  | 1.35                    | 3.24   | 1.17                    |
| 30                                     | 4.81   | 2.40                    | 5.6  | 2.18                    | 3.43   | 2.56                    |

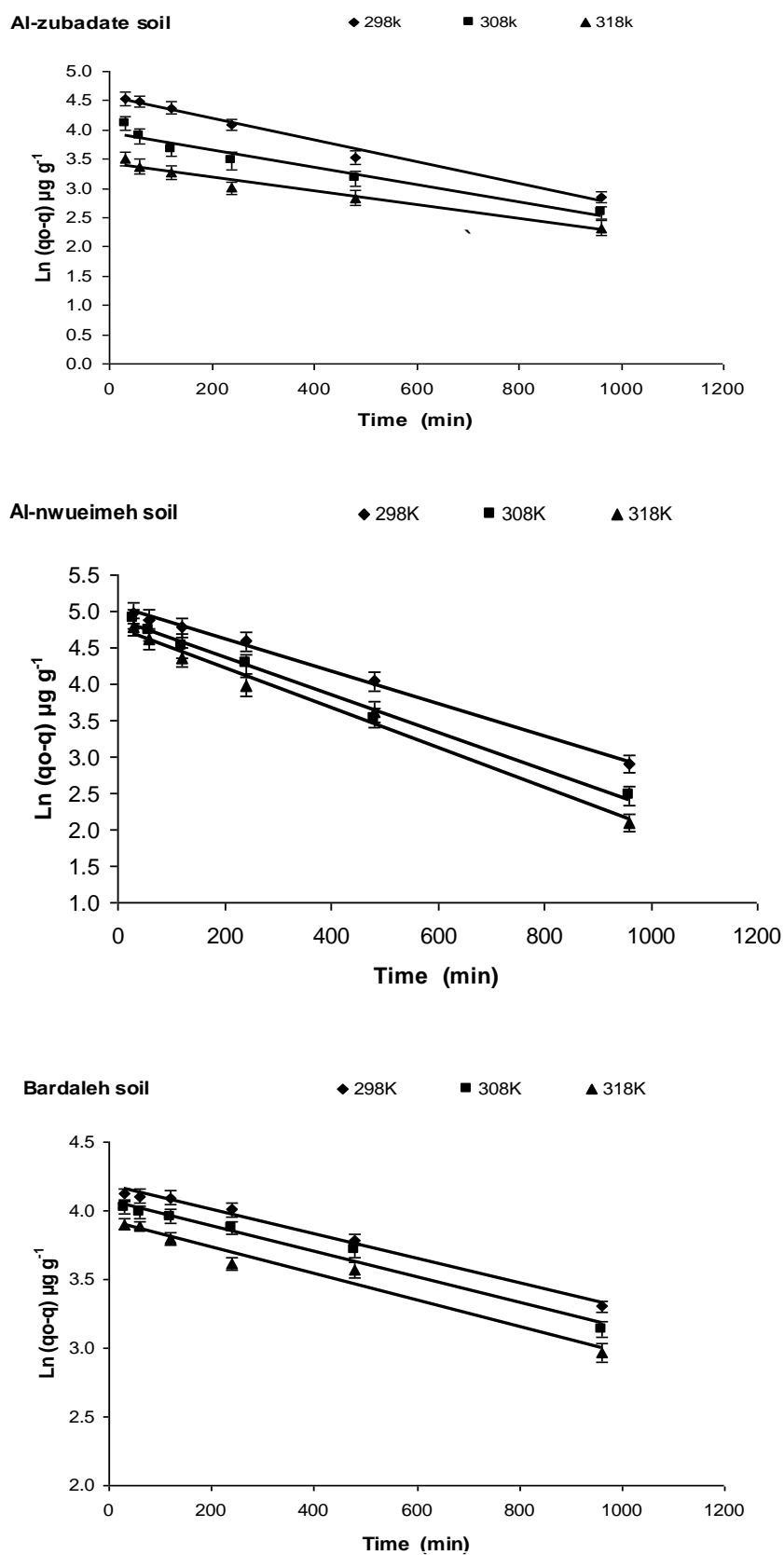
**Table 4:** Thermodynamic parameters of  $\beta$ -endosulfan sorption on the three studied soils at two  $\beta$ -endosulfan initial concentrations

| Initial Conc.<br>mg L <sup>-1</sup> | Ea<br>kJ mol <sup>-1</sup> | $\Delta G^\#$<br>(kJ mol <sup>-1</sup> ) | $\Delta S^\#$<br>J mol <sup>-1</sup> | $\Delta H^\#$<br>(kJ mol <sup>-1</sup> ) |
|-------------------------------------|----------------------------|--|--------------------------------------|--|
| <b>Al-Nuweimeh soil</b>             |                            |  |                                      |  |
| 15                                  | 22.47±0.04                 | 87.79±0.002                              | -22.74±3.88                          | 19.99±0.001                              |
| 30                                  | 21.25±0.04                 | 87.59±0.42                               | -23.08±0.69                          | 18.77±0.21                               |
| <b>Al-Zubadate soil</b>             |                            |  |                                      |  |
| 15                                  | 20.67±0.002                | 87.22±0.004                              | -23.15±7.14                          | 18.19±0.002                              |
| 30                                  | 18.57±0.002                | 86.99±0.004                              | -23.77±7.71                          | 16.10±.002                               |
| <b>Bardaleh soil</b>                |                            |  |                                      |  |
| 15                                  | 38.18±0.004                | 89.61±0.008                              | -17.88±0.014                         | 36.28±0.004                              |
| 30                                  | 38.07±0.001                | 89.34±0.0002                             | -18.02±0.38                          | 35.60±0.0001                             |



*Fig. 1:* Macroscopic sorption of  $\beta$ -endosulfan sorbed on three studied soils at 298K 546 and two different initial  $\beta$ -endosulfan concentrations vs. time





**Fig. 2:** First order equation plots of  $\beta$ -endosulfan sorption on the three studied soils at 3 temperatures and  $30 \text{ mg ml}^{-1}$  initial  $\beta$ -endosulfan concentration

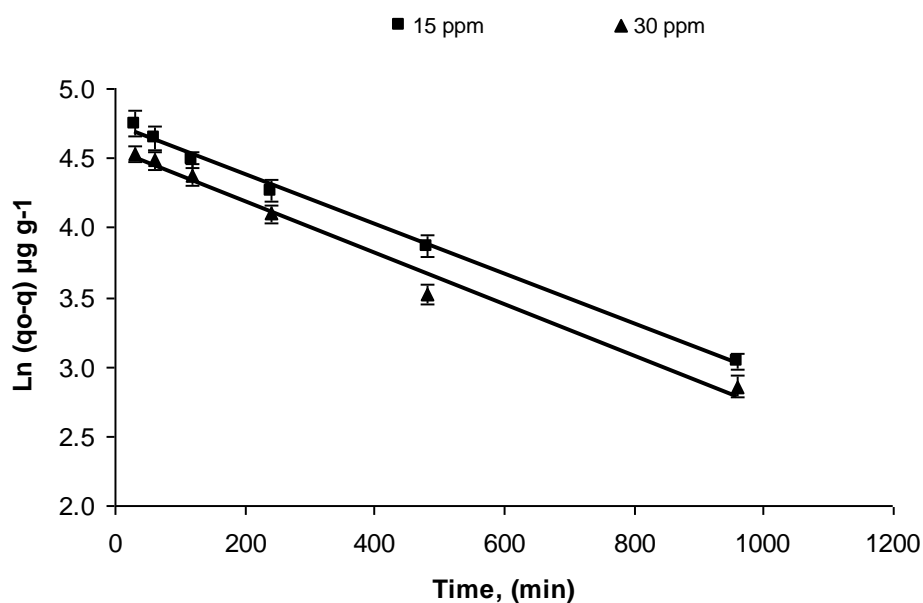


Fig. 3: Parallel relationship of the first order kinetic model with Al-Zubeidat soils at 298K and changing initial concentration while all other reaction parameters remained constant

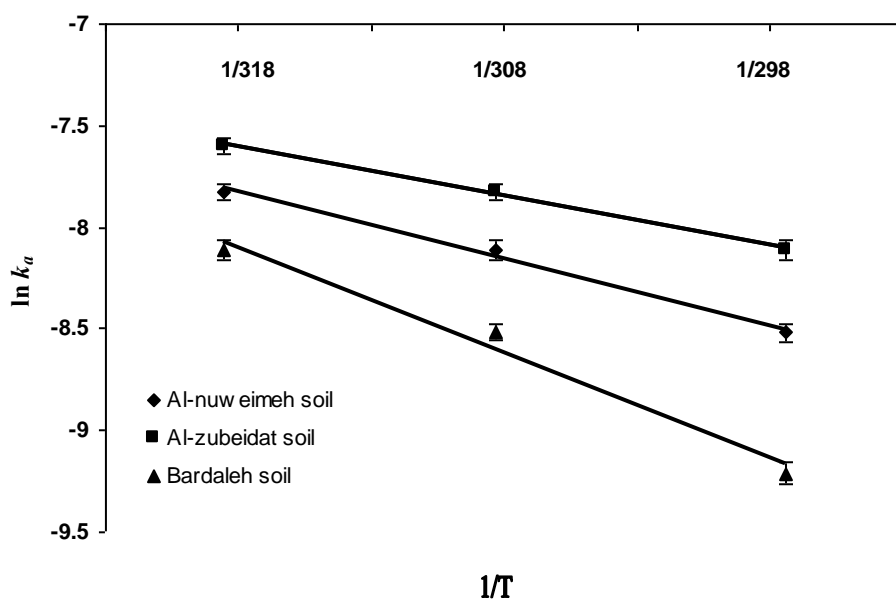


Fig. 4: Arrhenius plots of  $\beta$ -endosulfan sorption for three studied soils in Jordan Valley at three different temperatures



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## Studies on Alkyd Cross Linkable Acrylic Hybrid Emulsion for Coating Application

By Nilutpal Phukon, Tirthankar Jana & Tapan Kumar Dhar

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**Keywords:** *alkyd cross linkable emulsion, soya bean oil, emulsion polymerization, acid neutralized alkyd dispersion.*

**GJSFR-B Classification:** FOR Code: 039999



STUDIES ON ALKYD CROSS LINKABLE ACRYLIC HYBRID EMULSION FOR COATING APPLICATION

*Strictly as per the compliance and regulations of:*



RESEARCH | DIVERSITY | ETHICS

# Studies on Alkyd Cross Linkable Acrylic Hybrid Emulsion for Coating Application

Nilutpal Phukon <sup>α</sup>, Tirthankar Jana <sup>σ</sup> & Tapan Kumar Dhar <sup>ρ</sup>

**Abstract-** A series of alkyd with different oil length, acid and hydroxyl value were synthesized and used for cross linkable alkyd-acrylic hybrid emulsion via free radical polymerization using seeded semi continuous emulsion polymerization technique. The efficiency of different alkyds for stabilization during emulsion polymerization as well as emulsion stability and performance were studied. The stabilization of such type of emulsion was due to partial grafting of acrylic onto the unsaturated part of fatty acid in alkyd. The alkyd stabilized acrylic latex film was characterized by FTIR spectroscopy, thermal analysis and SEM study. The performance of such alkyd stabilized acrylic latex was also compared against acrylic emulsion with respect to drying, adhesion, flexibility and water repellency.

**Keywords:** alkyd cross linkable emulsion, soya bean oil, emulsion polymerization, acid neutralized alkyd dispersion.

## I. INTRODUCTION

Regulation of volatile organic compounds (VOC), incorporation of biomaterials, demand for long lasting sustainable high performance water based coating are the recent research trend in the field of organic coating. To enhance the performance of acrylic latex, alkyds are incorporated in the acrylic latex system as alkyd impart flexibility and acrylics have excellent durability and toughness properties [1]. A suitable combination of alkyd and acrylic may perform faster drying through crosslinking of acrylics and alkyd by auto oxidation process. However, it is very difficult to incorporate alkyd into acrylic system by emulsion polymerization to form alkyd – acrylic hybrid polymer [2]. Etham et. al. reported the modification of soya bean oil by sequential amidation and acrylation in the terminal double bonds which was used as co-monomer in acrylic emulsion polymerization [3]. Several efforts have been made via mini emulsion polymerization technique for incorporation of alkyd into acrylic phase [4 - 7]. It was also reported that the alkyd and acrylic polymer can be blended by melt condensation reaction [8]. Nabuurs et.al. reported the total conversion of acrylic monomers in presence of colloidal alkyd droplets [9]. Other than synthesis techniques, the performance of the coating film have also been enhanced by using allylic fatty acid derivative (AFAD) and an acrylic fatty acid derivative (AcFAD) by crosslinking process as both are reactive coalescing agents and co monomers [10]. Morenoa

et.al. reported the modification of oleic acid and linoleic acid by methyl methacrylate to form methacrylated oleic acid and methacrylated linoleic acid by grafting [11]. It has also been reported the emulsion polymer of methyl methacrylate modified soya bean oil formed by emulsion polymerization technique [12]. A series of work have been reported on acrylated alkyd emulsion formed by grafting of acrylic polymer from different monomers like methyl methacrylate, butyl acrylate, styrene, acrylic acid onto different alkyds based on linseed/soya bean oil followed by mini emulsion polymerization [13-18]. However, emulsion polymerization is an effective technique to synthesise hybrid latex and alkyd may be used as emulsifier after proper modification and acrylic monomers can be polymerized in presence of colloidal alkyd droplets.

In the present study, specially designed water dispersible soya bean oil fatty acid based alkyd resin having low molecular weight, low poly disparity with a balanced hydrophilic and hydrophobic structure was synthesized and used for the synthesis of alkyd-acrylic hybrid emulsion by free radical seeded semi continuous emulsion polymerization technique. To optimize the efficiency of alkyd in emulsion polymerization as well as hybrid emulsion stability, alkyds at different percentage of fatty acid, hydroxyl value and acid value were evaluated. The performance of such alkyd stabilized acrylic emulsion have also been evaluated and compared against conventional surfactant based acrylic emulsion.

## II. EXPERIMENTAL

### a) Materials

Commercial grade monomers, methyl methacrylate (MMA) (Gujarat State Fertilizers & Chemicals Ltd., India), n - butyl acrylate (BA) (LG Chem. Ltd. Korea) were used as received. Sodium lauryl sulphate (SLS) (Fisher Scientific, India) was used as surfactant. A water soluble initiator, Potassium persulfate (PPS) (Calibre Chemicals Pvt. Limited, India) was used for emulsion polymerization. Organic initiator Tert-butyl hydro peroxide (TBHP) (70% solution) (Spectrochem Pvt Ltd, India) and Sodium meta bisulfite (SMBS) (Fluka, USA) were used as redox pair initiator. Sodium bicarbonate (SBC) (Darshan Chemicals, India) was used as buffer to control pH during reaction. Distilled water was used in all steps of the processes. Liquid ammonia (28% aq. Solution), (Indian Scientific

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Enterprises, India) was used as such for neutralization. Soya bean oil fatty acid (SOFA) (Kargill Ltd., India), Tri methylol propane (TMP) (Perstrop Ltd., Sweden), Pentaerythritol (PE) (Perstrop Ltd., Saudi Arabia), Phthalic anhydride (Thirumalai Chemicals Ltd. India), Benzoic acid (BZA) (IG Petrochemicals Ltd., India) all these materials used for the synthesis of resin are of commercial grade and used as received without any further purification.

#### b) Preparation of Alkyd and Alkyd Dispersion

The formulation of different alkyds and their properties are presented in Table 1. For preparation of

different alkyds, fatty acid, polyols, poly acid and monobasic acid were charged in a four necked round bottom flask fitted with a mechanical stirrer, a condenser attached with a Dean and Stark apparatus and a thermometer. The reaction was carried out at 160o C to 220o C and continued to achieve the desired final acid value.

**Table 1:** Composition of Different Alkyd Resins and their Properties

| Resin Code | Resin Composition |       |      |       |      | Formulation constant |                           | Physical Properties of Resin |      |      |               |
|------------|-------------------|-------|------|-------|------|----------------------|---------------------------|------------------------------|------|------|---------------|
|            | SOFA              | TMP   | PE   | PA    | BZA  | Oil length, %        | Hydroxyl value, mg KOH/gm | Acid value, mg KOH/gm        | Mw   | PDI  | Viscosity, cp |
| Alk 01     | 42.5              | 22.2  | 5.5  | 24.3  | 5.5  | 53                   | 78                        | 30                           | 3793 | 1.59 | 54983         |
| Alk 02     | 35                | 25    | 5    | 29    | 6    | 45                   | 78                        | 28                           | 5012 | 2.09 | 2390000       |
| Alk 03     | 51                | 21    | 5    | 19    | 4    | 62                   | 78                        | 26                           | 2812 | 1.53 | 11550         |
| Alk 04     | 42.5              | 22.2  | 5.5  | 24.3  | 5.5  | 53                   | 78                        | 21                           | 6915 | 2.83 | 350000        |
| Alk 05     | 42.5              | 22.2  | 5.5  | 24.3  | 5.5  | 53                   | 78                        | 41                           | 2840 | 1.64 | 110370        |
| Alk 06     | 42.24             | 20.55 | 5.55 | 26.66 | 5    | 53                   | 57                        | 31                           | 3160 | 1.75 | 103430        |
| Alk 07     | 42.22             | 22.22 | 6.66 | 23.35 | 5.55 | 53                   | 103                       | 30                           | 3000 | 1.65 | 94000         |

For preparation of alkyd dispersion, 50 gm. of alkyd, 47.9 gm. of D.M. water and 2.5 gm of ammonia (28 percent in aqueous solution) were taken in a container and mixed under high speed dispersion at rpm of 900-1000 for 1hr.

#### c) Use of Alkyd Dispersion in the Preparation of Acrylic Emulsion

The water dispersible alkyd was used as a stabilizer for the preparation of acrylic emulsion (O/W). Three different types of latex (Type I, II and III) were designed to identify the effectiveness of alkyd as emulsion stabilizer. Type I: Surfactant used in nucleation process and pre-emulsion stabilization. Type II: Initiation started with surfactant nucleated process while alkyd dispersion was used in the pre-emulsion. Type III: Alkyd dispersion was used in nucleation process as well as pre-emulsion stabilization. The formulation recipe for all different types of emulsion is presented in Table 2. The emulsion polymerization was carried out in a three necked 2 litre capacity reactor equipped with stirrer, reflux condenser, feeding inlet and thermometer. Initially, the reactor was charged and equivalent quantity of monomers i.e. methyl methacrylate and n-butyl acrylate. After initial charging, the reactor was heated up to 800 C followed by addition of initiator to start the initiation. After initiation, 5% pre-emulsion, 6% initiator PPS (5% wt.

solution) and 6% SBC (6% wt. solution) solution w.r.t. monomer were charged into the reactor under continuous stirring. After initiation and continuous feeding of pre emulsion was done for 4 hrs. Alkyd was used in the reaction medium during synthesis of Type II and III emulsion. The completion of reaction was monitored after measurement of solid content of the reaction mixture. Finally, TBHP and SMBS (8% wt. solution) were added in the reactor to deactivate any unreacted monomer present in the reactor.



Table 2: Formulation Recipe of Different Latex and their Properties

| Latex/Emulsion type | Pre emulsion composition |                               | Reactor charge   |                               | Emulsion Properties |        |
|---------------------|--------------------------|-------------------------------|------------------|-------------------------------|---------------------|--------|
|                     | Ionic surfactant         | Alkyd dispersion (Alkyd Type) | Ionic surfactant | Alkyd dispersion (Alkyd Type) | Solid, %            | Tg, °C |
| AL/Type I           | 1.13                     | 0                             | 0.34             | 0                             | 48.9                | 12     |
| ASAL01/Type II      | 0                        | 2.3(Alk03)                    | 0.34             | 0                             | 44.8                | 10.77  |
| ASAL02/Type II      | 0                        | 2.3(Alk06)                    | 0.34             | 0                             | 44.9                | 11.8   |
| ASAL03/Type II      | 0                        | 2.3(Alk05)                    | 0.34             | 0                             | 44.8                | 10.92  |
| ASAL04/Type II      | 0                        | 2.3(Alk02)                    | 0.34             | 0                             | 44.9                | 11.57  |
| ASAL05/Type III     | 0                        | 2.3(Alk01)                    | 0                | 2.3(Alk01)                    | 44.9                | 10.42  |
| ASAL06/Type II      | 0                        | 2.3(Alk01)                    | 0.34             | 0                             | 44.9                | 10.54  |

#### d) Film Preparation

The newly synthesised acrylic emulsion samples were coated on mild steel and glass panels using box coater of 50 microns. The coated panels were cured at ambient temperature (300C) for 48 hrs, for complete maturation of the films before testing.

### III. TEST METHODS, CALCULATIONS AND POLYMER CHARACTERIZATION

#### a) Scratch Hardness Test as per BS 3900 Part E2

Scratch resistance of a paint film is a measure of resistance to penetration by scratching with a needle. In this test, a scratching needle of 1mm diameter with hemispherical hardened steel tip, along with different specified load is applied over the needle and scratched the cured coating surface of the test paint. The loading on the needle is increased until the needle penetrates through the coating to the substrate. The minimum weight, which penetrates the coating, is recorded as the scratch resistance.

#### b) Flexibility as per ASTM D522

The test is carried out by bending the coated metal panel over round cylindrical mandrels of varying diameters, the film being bend on the outside. After bending, the coating is examined for cracking, flaking etc. The smaller the diameter of mandrel at which cracks appear in the film, the better is the flexibility of the coating material.

#### c) Adhesion as per ASTM D 3359-95a

The cross cut adhesion test was performed on the samples coated on mild steel panels and cut into squares of 1x1 mm, adhesive tape was applied to the cross cut area firmly rubbed with a fingertip and removed after 2 min. the number of squares intact gives a measure of adhesion of the sample.

Adhesion (%) = Number of squares intact in the tape applied area / Total number of squares in the tape applied area x 100

#### d) Percentage Conversion

Percentage Conversion (% NVM) was measured by gravimetric analysis after completion of reaction. 1 g of latex sample was weighed in a steel lid (Wi), dried it for 1 h at 120 °C and again weighted (Wd). The final non-volatile matter (% NVM) was estimated by the following equation.

$$\% \text{ NVM} = \frac{W_d}{W_i} \times 100$$

#### e) Emulsion Stability

Shelf life stability was measured by placing 30 ml of the emulsion in a capped glass vial and kept at ambient temperature (30°C). The time needed for separation in the form of two layers to appear was monitored.

#### f) SEM Study

Specimen for SEM micrographs were prepared by sputter-coated with gold and micrograph images were taken under low vacuum mode, 10 kV voltages within a JEOL JSM 8360 SEM device.

#### g) DSC Analysis

The glass transition temperature (Tg) of different latex film sample was measured by differential scanning calorimetry (DSC), Perkin Elmer DSC 4000 model. The latex film sample in the range of 3 – 5 mg was held within a dedicated sample pan. The sample was scanned between - 60 °C to 120 °C at heating rate of 10 °C/min and cooling rate of 10 °C/min for two cycles under nitrogen atmosphere and the heat flux versus temperature data was recorded.

#### h) GPC Study

The relative values of weight average (Mw) and number average (Mn) molecular weights of all the alkyd resins were measured by Gel permeation chromatography (Waters). Tetrahydrofuran was used as eluting solvent. The instrument contains three poly styragel column (1000A<sup>0</sup>, 500A<sup>0</sup>, 100A<sup>0</sup>). Flow rate was

0.3 ml/min. calibration was made using polystyrene standards.

i) *Viscosity*

Viscosity of different alkyd resins were measured at 25 °C using modular compact Rheometer (MCR 102) over the range of shear rate 0 – 60 s<sup>-1</sup>. Viscosity was reported at 20.5 s<sup>-1</sup> and 2.5 s<sup>-1</sup> in the case of high viscous alkyd like Alk-02 and Alk-03.

j) *Contact Angle*

The emulsions were applied by brush on glass panels and cured for 7 days at room temperature (30°C). The static contact angle was measured by dropping water droplet on the surface of the dry coated film.

k) *Water Resistance*

The emulsion was applied on glass panel at a thickness of 200 micron and dried for 7 days at 30°C. After proper drying, the coated panels were immersed in water and the film defects were monitored at different time intervals.

l) *Alkali Resistance*

The emulsion was applied on glass panel at a thickness of 200 micron and dried for 7 days at 30°C. After proper drying, the coated panels were immersed in 0.1(N) aqueous NaOH solution and the film defects were monitored at different time intervals.

m) *Solvent swelling*

The coating applied on glass panels at a film thickness of 200 micron and dried for 7 days at 30°C. After drying aromatic solvent like xylene was dropped on the surface of the film and swelling was monitored.

## IV. RESULTS AND DISCUSSION

a) *Effect of Emulsion Stability on the alkyd oil length, acid value and hydroxyl value*

The stability of hybrid emulsion is a very important characteristic for storage of emulsion. The synthesised hybrid emulsions were kept in airtight bottle and stored at room temperature for a period of 12 months. The variation of emulsion stability with oil length of the alkyd is presented in Fig. 1.

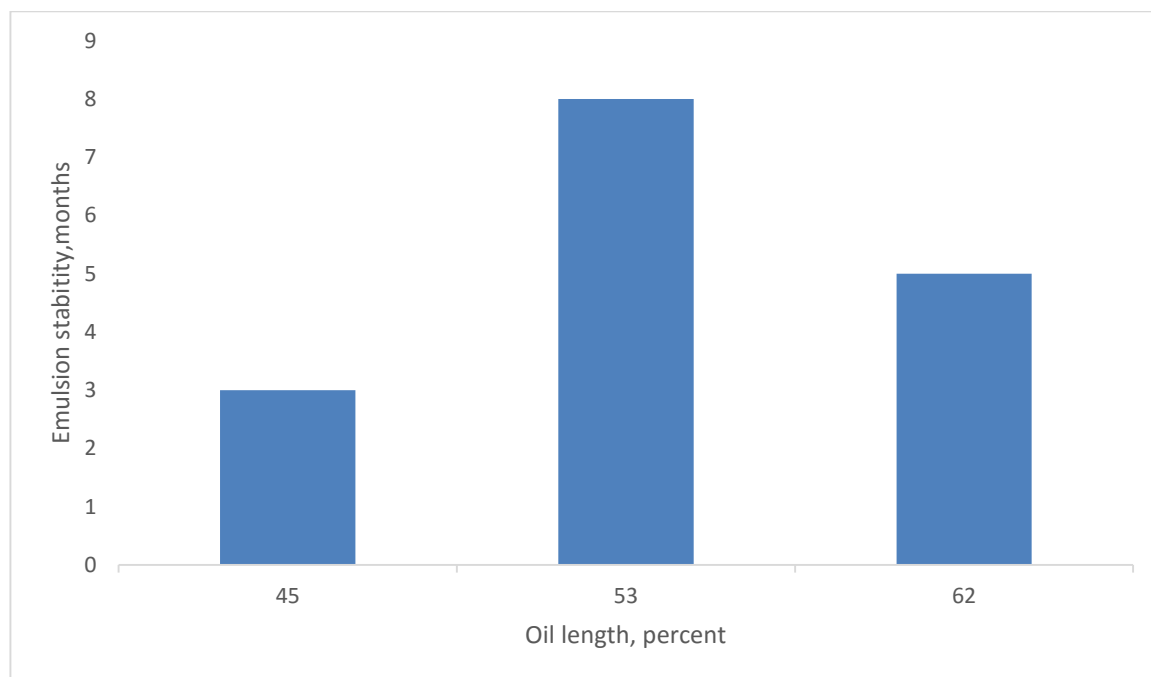
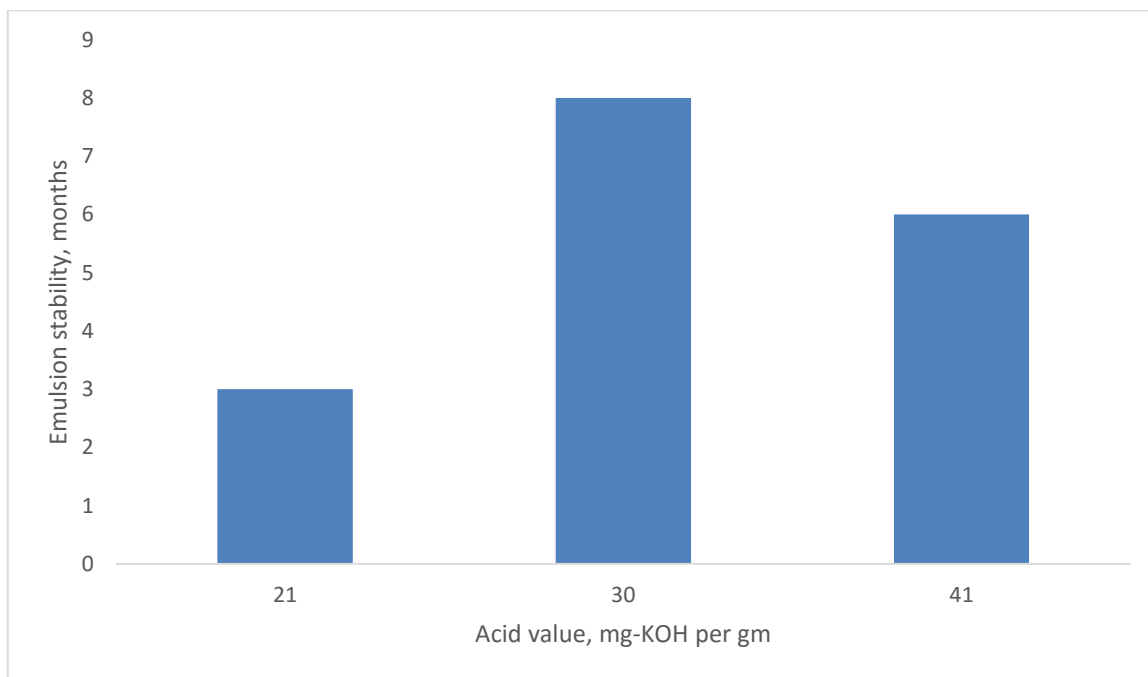


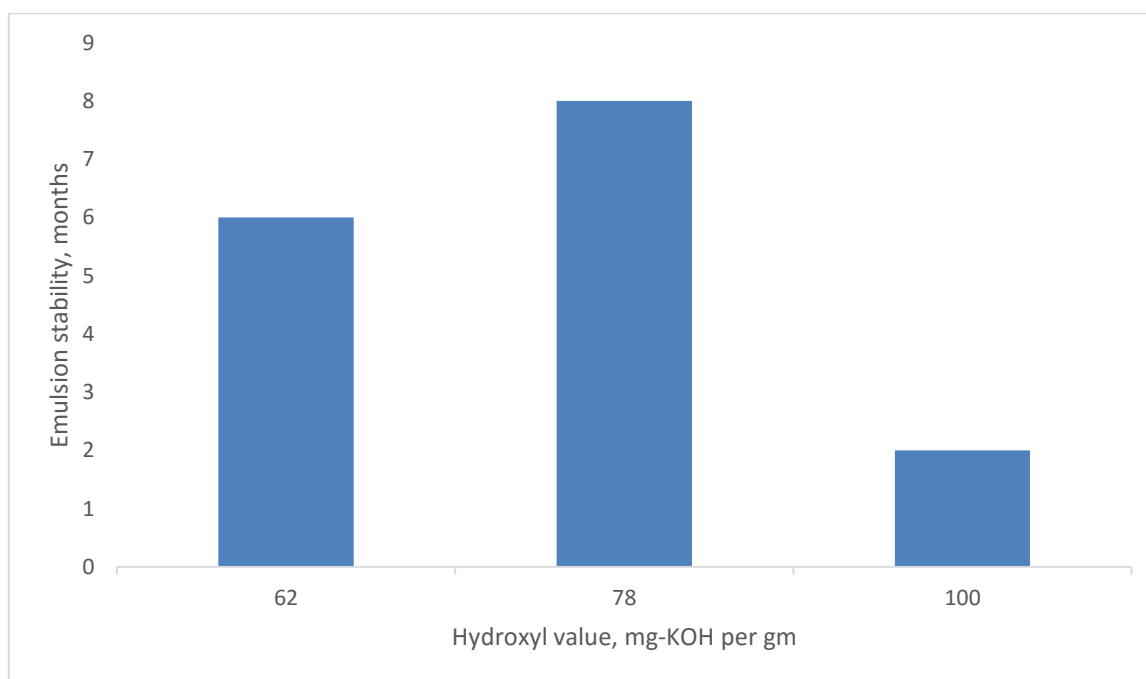
Fig. 1: Effect of emulsion stability on oil length of alkyd resin

The dispersion of alkyd after neutralization of the acid functional groups, depends on the molecular weight of the alkyd. The dispersion stability of emulsion using 45 percent oil length alkyd is not good due to high molecular weight compared to 53 and 62 percent oil length alkyd. However, with increase in oil length of the alkyd, the hydrophobic nature of alkyd mainly control the emulsion stability [19]. As a result, although the molecular weight of 62 percent oil length alkyd is low,

but the hybrid emulsion stability with this alkyd inferior due to the presence of higher percentage of oil in the alkyd. The effect of hybrid emulsion stability on acid value and hydroxyl value of alkyd resin are presented in Fig. 2 and 3 respectively.



*Fig. 2:* Effect of emulsion stability on Acid value of alkyd resin



*Fig. 3:* Effect of emulsion stability on Hydroxyl value of alkyd resin

The increase of acid and hydroxyl value of any alkyd resin, increases the polarity of the resin. The acid functional groups of alkyd resin control water solubility after neutralization with any base. During nucleation and stabilization of growing particles in emulsion polymerization, the specially designed alkyd molecules

(i.e. a balanced hydrophilic and hydrophobic structure and optimum molecular weight) were dynamically arranged on the particle surface and helps for the growth of particles without affecting the conversion of monomer [20]. The polarity of alkyd chain controls the dynamic orientation of alkyd chain. However, the

surfactant nucleated latexes in combination with alkyd showed comparatively better emulsion stability in terms of phase separation as compared to the alkyd nucleated surfactant free latex due to a balanced polar structure. The hybrid emulsion containing alkyd resin having 53 percent oil length, 30 mg-KOH per gm acid value and 78 mg-KOH per gm is an optimum combination to achieve maximum stability.

*b) Effect of alkyd resin on emulsion Polymerization*

The dispersion of alkyd helps for stabilization of monomer pre emulsions and finally, converted into acrylic polymer through free radical emulsion polymerization technique. It has been found that the initiation of alkyd stabilized monomer pre emulsion for free radical emulsion polymerization was quite slow compared to conventional ionic surfactant stabilized system. The higher concentration of unsaturated fatty

acids retards the rate of polymerization. Grafting can occur on the acrylic chain by free radical chain transfer mechanism [21, 22]. This indicates the participation of alkyd in the reaction kinetics after capturing some radicals. This slow induction does not affect the overall conversion in the polymer chain. From gravimetric analysis, it was found that nearly 100 percent conversion achieved without any unwanted coagulum formation using 1.15 and 2.3 percent of alkyd (Type II & Type III) in the emulsion polymerization. Similar observation was also reported earlier by mini emulsion polymerization [17].

*c) Comparative study on the coating properties of pure acrylic and alkyd acrylic hybrid emulsion*

The coating properties of pure acrylic and alkyd acrylic hybrid emulsion (ASAL06/Type II) is presented in Table 3.

*Table 3:* Comparative evaluation of pure acrylic and alkyd acrylic hybrid emulsion

|  | Pure Acrylic         | Alkyd Acrylic Hybrid |
|--|----------------------|----------------------|
| <b>Emulsion Properties</b>                         |                      |                      |
| Appearance   | Milky white emulsion | Off white emulsion   |
| Non - volatile content, Percent                    | 45.21                | 44.9                 |
| p <sup>H</sup>                                     | 7.8                  | 8.2                  |
| Viscosity in Ford Cup B4@ 30°C, Second             | 44                   | 42                   |
| <b>Dry Film Properties</b>                         |                      |                      |
| Drying time, minutes                               | 60                   | 70                   |
| Scratch hardness, gm                               | 1100                 | 1000                 |
| Adhesion (cross hatch), %                          | 95                   | 100                  |
| Flexibility, ¼ inch                                | Slight inferior      | Good                 |
| Water resistance, hrs                              | 52                   | 48                   |
| Alkali resistance, hrs                             | 40                   | 32                   |
| Glass Transition Temperature (T <sub>g</sub> , °C) | 12                   | 10.5                 |
| Contact angle, Degree                              | 88.5                 | 100                  |
| Swelling resistance , Rating 1 poor and 10 best    | 5                    | 7                    |

The drying time of hybrid acrylic film is almost comparable with pure acrylic although alkyd have been incorporated in hybrid emulsion. This is due to grafting of acrylic monomers onto the unsaturated part of fatty acid. It has been observed that the hybrid emulsion film have very good adhesion and flexibility compared to hardness due to the presence of flexible alkyd component. This indicates excellent compatibility between alkyd and acrylate phases in hybrid emulsion [19]. It has been found that the swelling resistance and contact angle increases in the alkyd stabilised acrylic resin. This indicates the existence of post oxidative crosslinking in the alkyd stabilized latex system which reduces the surface swelling tendency [19]. However, it has also been found that the resistance to solvent swelling was better for high oil length alkyd which was also an indication of effective grafting. It has been found that the alkyd emulsion stability of the combination like ASAL01 with Alk-03, ASAL02 with Alk-06 and ASAL03 with Alk-05 were good and the contact angle were more than 80 for all cases. However, it has been found that

the reduction of hydroxyl value decreases swelling and hydrophobicity of latex film. This is due to lower molecular integration or association in the hybrid emulsion. The contact angle of surfactant containing alkyd stabilized latex was higher than surfactant free latex ASAL05. This is an indication of surface modification of latex by alkyd molecules. It has also been found that a combination of higher dose of alkyd with zero surfactant does not enhance the contact angle. The effect of surface swelling on different hybrid latex and method of preparation is presented in Fig. 4. This was due to the generation of poor nucleation but effective acrylic grafting onto alkyd chain. The poor nucleation and effective alkyd grafting produced improper crosslinking. Out of these three, Alk-06 was comparatively more hydrophobic. Hence, this hydrophobicity enhancement depended on the availability of hydrophobic soybean fatty acid, molecular integration into acrylic phases and stability after latex synthesis.

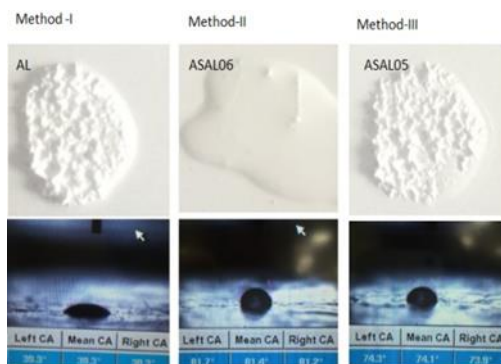


Fig. 4: Effect of different preparation methods on the swelling of different emulsion film

#### d) FTIR Analysis

The FTIR spectra of pure acrylic and alkyd acrylic hybrid resin are shown in Fig. 5a and 5b

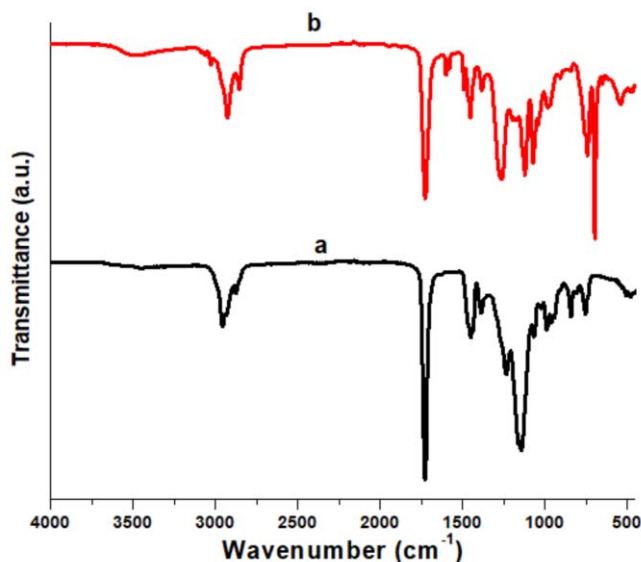


Fig. 5: FTIR spectra of pure acrylic resin 5(a) and alkyd acrylic hybrid resin 5(b)

#### e) SEM Study

The scanning electron micrograph of surfactant stabilized acrylic latex and alkyd stabilized acrylic latex are presented in Fig. 6

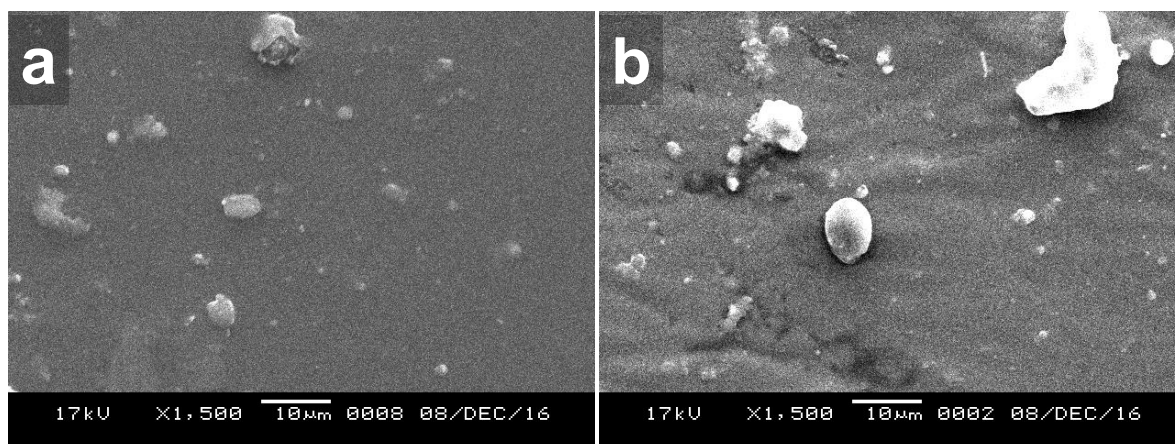


Fig. 6a: SEM of acrylic latex

Fig. 6b: SEM of alkyd stabilized acrylic latex

respectively. The presence of sharp peak at  $1723\text{ cm}^{-1}$  indicate the ester group of acrylate and alkyd resin. In Fig. 5 b, the bands at around  $1129\text{ cm}^{-1}$  and  $1273\text{ cm}^{-1}$  are assigned to stretching and bending frequency of C-O-C, CH bond respectively. The broad band at  $3453\text{ cm}^{-1}$  in Fig. 5b is due to the presence of free O-H group of alkyd resin. The absorption bands in the region of  $2925\text{ cm}^{-1}$  to  $2850\text{ cm}^{-1}$  is due to C-H stretching of  $-\text{CH}_3$  and  $-\text{CH}_2$  groups respectively [23, 24]. In Fig 5b, the CH out of plane bending frequencies at  $729\text{ cm}^{-1}$  suggest the presence of aromatic residues in the resin and the stretching frequencies at  $1580\text{ cm}^{-1}$  and  $1607\text{ cm}^{-1}$  is due to residual C=C in alkyd resin. However, the absence of absorption bands in this region (Fig.5a) indicate that all monomers are converted into acrylic polymer.



The surface morphology of alkyd stabilized acrylic latex (Fig. 6b) was more non uniform than that of surfactant stabilized acrylic latex (Fig. 6a). Pitting and surface roughness were observed in the alkyd stabilized acrylic latex film. On stabilization of acrylic by alkyd chain, the surface of the acrylic film was masked with the alkyd chain and due to uneven masking the roughness of the film were formed. Similar type of observation has also been reported after grafting of acrylic monomer [25, 26]. So the increase of surface roughness in the alkyd stabilized acrylic latex was an indication of surface grafting. During drying process, the oxidative polymerization of alkyd creates shrinkage on alkyd stabilized acrylic film surface and surface roughness is formed.

#### f) DSC Analysis

The DSC thermogram of different alkyd stabilized acrylic and without alkyd stabilized acrylic film

is presented in Fig. 7. In DSC thermogram, the single heat flow indicates that there was no multiple domains of polymer structure was generated in the alkyd stabilized acrylic system. The onset of heat flow were takes place little earlier as compared to pure acrylic emulsion (AL) [Fig. 7 a]. It was reported that alkyd after grafting onto acrylic structure changed the domains of alkyd in such a way that there was no separate phase of alkyd in the acrylic structure [14]. It is also observed that after incorporation of alkyd into acrylic matrix, the glass transition temperature ( $T_g$ ) of all alkyd stabilized acrylic hybrid emulsion is not affected [Fig. 7 b, c and d]. Because the unsaturation present in fatty acid part was partly participated in the grafting of acrylic onto alkyd structure. After grafting the pendent unsaturation was used for post crosslinking which enhanced the solvent resistance property.

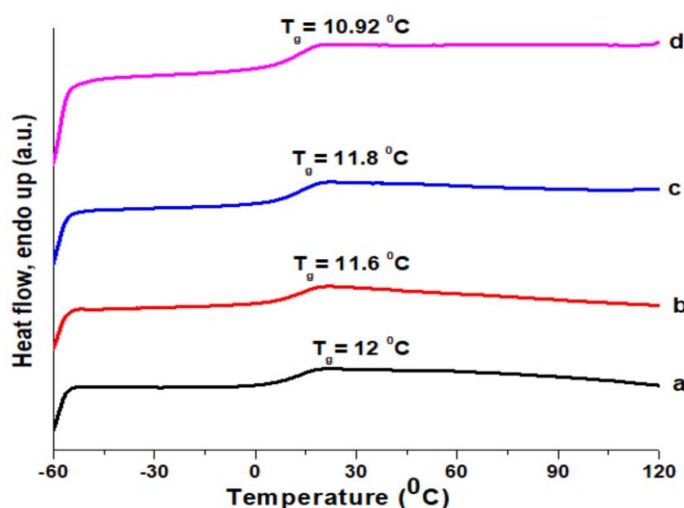


Fig. 7: DSC Thermogram of Pure acrylic (a), 45% oil length alkyd modified acrylic (b), 53% oil length alkyd modified acrylic (c), 62% oil length alkyd modified acrylic (d)

## V. CONCLUSION

The present study shows that stable acrylic o/w emulsion is formed with alkyd dispersion by semi-continuous emulsion polymerization. The optimum conversion of acrylic monomer into acrylic polymer indicates that there is no retardation of monomer transportation from droplets to the locus of the polymerization. The increase in adhesion and flexibility, surface swelling resistance and stability of alkyd stabilized acrylic emulsion indicates that there is an interaction between acrylic resin and alkyd structure. The medium oil length alkyd with acid value and hydroxyl value in the range of 30 and 78 mg KOH per gm respectively and low molecular weight is effective for the stability of alkyd stabilised acrylic emulsion. The higher water repellence and solvent swelling resistance of the latex film are due to greater interpenetration and

entanglement of the two phases. This is a novel technique for preparation of stable pre-emulsion without using any surfactant only by using soya fatty acid based water dispersible alkyd.

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# A Study on the Chemical Separation of *Ficus Rumphii* Blume Extract

By Md. Islamul Haque & Md. Mohsin Uddin Azad

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**Abstract-** *Ficus rumphii* Blume (Family: Moraceae), commonly known as “Gaiaswat” or “Pakar”, is widely used in Bangladeshi traditional medicine to treat various diseases including asthma, kill worms and in snake bite<sup>1</sup>. The present study was undertaken to separate the solution that is responsible for the antimicrobial agents whom we will further research later.

**Keywords:** *ficus rumphii*, *antimicrobial*.

**GJSFR-B Classification:** FOR Code: 259999



*Strictly as per the compliance and regulations of:*



# A Study on the Chemical Separation of *Ficus Rumphii* Blume Extract

Md. Islamul Haque <sup>α</sup> & Md. Mohsin Uddin Azad <sup>σ</sup>

**Abstract-** *Ficus rumphii* Blume (Family: Moraceae), commonly known as “Gaiaswat” or “Pakar”, is widely used in Bangladeshi traditional medicine to treat various diseases including asthma, kill worms and in snake bite<sup>1</sup>. The present study was undertaken to separate the solution that is responsible for the antimicrobial agents whom we will further research later.

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## I. INTRODUCTION

*Ficus rumphii* Blume (Family: Moraceae), commonly known as “Gaiaswat” or “Pakar”, is a tree basically found around north-east of Bangladesh. They are not only useful for their local use in cuts and wounds but also it has some medicinal use. This medicinal use is to cure asthma, killing worms and also it is used to detoxicate snake bite. Its bark is really a useful thing. It is also used in burning sensation, leucoderma, ulcers, leprosy, itching, biliousness and diseases of blood. The leaves boiled in oil form good applications for wounds and bruises<sup>2-4</sup>.

## II. PREPARATION OF EXTRACT

The bark of *Ficus rumphii* Blume was collected from Harinarayanpur, Purboabdalpur, Laksumpur & Modhupur village in the district of Kushtia of Bangladesh in June-July 2017 and identified by a plant taxonomist and voucher specimen has been deposited in Bangladesh National Herbarium.

The collected plants were separated from undesirable materials or plants or plant parts and then were washed several times with tap water to remove the adhering dirt. Then the stem barks was cut into small slices and were shade-dried for four week. The shade dried plant materials were dried in an electric oven at 40°C for about 24 hours when the pieces were almost dry. The dried plant materials were ground into a coarse powder with the help of a suitable grinder (Capacitor start motor, Wuhu motor factory, China). The powder was stored in an airtight container and kept in a cool, dark and dry place until extraction with solvent and hydro distillation.

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Fig. 1: *Ficus rumphii* Blume plant

## III. ISOLATION OF BARK ESSENTIAL OIL

The air-dried bark (200 g) of *F. rumphii* was subjected to hydro distillation for 3 hour using a Clevenger type apparatus. The oil was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and preserved in a sealed vial at 4°C until further analysis.

## IV. PRELIMINARY EXTRACTION FROM POWDERED PLANT MATERIALS

The powdered plant materials (1300 gm) was placed in different reagent bottles (number of bottles is 13 and each contains 100 gm of powder plant materials) and extracted with petroleum ether (b.p. 40-60°C), ethyl acetate (b.p 70-72°C) and methanol (b.p 63-65°C). The mixture was stirred by glass rod every day an hour. After 7 days the extract was filtered through a filter paper in several times. The residue remaining after extraction of above was further extracted at room temperature with ethyl acetate. The mixture was stirred by glass rod every day an hour. After 7 days the ethyl acetate extract was filtered through a filter paper in several times. The residue remaining after extraction of above was further extracted at room temperature with methanol. The mixture was stirred by glass rod every day an hour. After 7 days the ethyl acetate extract was filtered through a filter paper in several times. The solvent is added according to the polarity.

## V. INVESTIGATION OF EXTRACT

The bark ethyl acetate extract (10 gm) of *F. rumphii* was subjected to column chromatography for fractionation. The column was run with solvent of

increasing polarity. Starting with 100% petroleum ether, polarity of the solvent (mobile phase) was increasing gradually by adding increasing proportions of ethyl acetate and methanol. The subsequent elutes were collected in 100 ml flasks.

All the fractions from ethyl acetate extract were tested on TLC under UV light, in iodine chamber and by

vanillin-sulfuric acid spray. Similar fractions were tested depending on the TLC behavior and were classified into following groups, Group A (Fr. 1), Group B (Fr. 2), Group C (Fr. 3 - 5), Group D (Fr. 6-8), Group E (Fr. 9), Group F (Fr. 10-15), Group G (Fr. 16-20), Group H (Fr. 21-26).

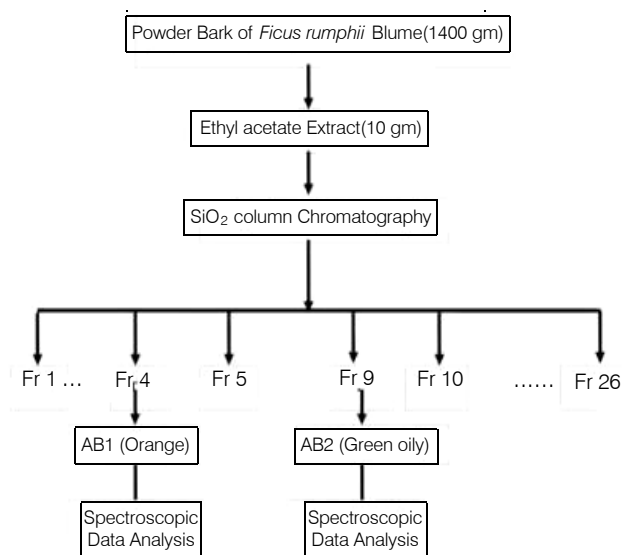
**Table 1:** Solvent ratio used in column chromatography of bark extract of *Ficus rumphii* Blume

| Solvent                        | Ratio | Volume (ml) | Fractions collected in the beaker |
|--------------------------------|-------|-------------|-----------------------------------|
| Petroleum ether                | 100 % | 100         | 1                                 |
| Petroleum ether: Ethyl acetate | 9 : 1 | 100         | 2                                 |
| Petroleum ether: Ethyl acetate | 8 : 2 | 100         | 3 - 5                             |
| Petroleum ether: Ethyl acetate | 7 : 3 | 100         | 6 - 8                             |
| Petroleum ether: Ethyl acetate | 6 : 4 | 100         | 9                                 |
| Petroleum ether: Ethyl acetate | 5 : 5 | 100         | 10                                |
| Petroleum ether: Ethyl acetate | 4 : 6 | 100         | 11                                |
| Petroleum ether: Ethyl acetate | 3 : 7 | 100         | 12                                |
| Petroleum ether: Ethyl acetate | 2 : 8 | 100         | 13                                |
| Petroleum ether: Ethyl acetate | 1 : 9 | 100         | 14                                |
| Ethyl acetate                  | 100 % | 100         | 15                                |
| Ethyl acetate: Methanol        | 9 : 1 | 100         | 16                                |
| Ethyl acetate: Methanol        | 8 : 2 | 100         | 17                                |
| Ethyl acetate: Methanol        | 7 : 3 | 100         | 18                                |
| Ethyl acetate: Methanol        | 6 : 4 | 100         | 19                                |
| Ethyl acetate: Methanol        | 5 : 5 | 100         | 20                                |
| Ethyl acetate: Methanol        | 4 : 6 | 100         | 21                                |
| Ethyl acetate: Methanol        | 3 : 7 | 100         | 22                                |
| Ethyl acetate: Methanol        | 2 : 8 | 100         | 23                                |
| Ethyl acetate: Methanol        | 1 : 9 | 100         | 24                                |
| Methanol                       | 100 % | 200         | 25- 26                            |

## VI. RESULT

Based on bio-assay, the ethyl acetate bark extract was selected for the purification of the compounds. The bark extract (12 gm) was subjected to column chromatography over silica gel (230-400 mesh ASTM, Merck, Germany) to give 26 fractions. The two compounds obtained from the fraction 4 are designed as compound AB1 by TLC using solvent ratio of petroleum ether: ethyl acetate 8.75: 1.25. The one compound obtained from the fraction 9 is designed as compound AB2 by TLC using solvent ratio of petroleum ether: dichloromethane 8.3: 1.7.





**Diagram 1:** Scheme for Isolation and purification of compounds AB1 & AB2 Green oily from Extract of *Ficus rumphii* Blume bark



**Fig. 2:** Separation by column chromatography

## VII. DISCUSSION

The antimicrobial activity of the petroleum ether, ethyl acetate and methanol extracts will be carried out against various gram positive and gram-negative bacteria. The zone of inhibition formed against these microorganisms will be measured for the determination of the antibacterial efficacy of the different plant extracts of significant antibacterial potential against *Bacillus subtilis*, *Sarcina lutea*, *Escherichia coli* and *Pseudomonas* sp.

## ACKNOWLEDGEMENT

The authors are grateful to Islamic University, Kushtia and Bangladesh National Herbarium.

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# Synthesis and Chemistry of Bis-imidazole Derivatives: A Review

By Abdullah Elmorsy, A. M. S. Hebishy, Ahmed Elwahy & M. S. Abdelfattah

*Helwan University*

**Abstract-** Imidazole, an important heterocycle, having biological and therapeutic properties prompted various medicinal chemists to develop various synthetic methods to synthesize a large number of novel derivatives used in chemotherapy. This fascinating group of heterocyclic compounds has interesting biological activities such as antimicrobial, anticancer, antiviral, analgesic, anti-inflammatory, anthelmintic, anticonvulsant, antiulcer, and anti-allergic activity. Various bis-imidazole derivatives introduced a great contribution not only in the field of synthetic organic chemistry but also in the field of medicinal chemistry and chemotherapy. This article aims to review the work reported in the synthesis and chemistry of bis-imidazole derivatives during past years.

**Keywords:** *imidazole, heterocycle, bis-imidazole.*

**GJSFR-B Classification:** *FOR Code: 039999*



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# Synthesis and Chemistry of Bis-imidazole Derivatives: A Review

Abdullah Elmorsy <sup>α</sup>, A. M. S. Hebishy <sup>σ</sup>, Ahmed Elwahy <sup>ρ</sup> & M. S. Abdelfattah <sup>ω</sup>

**Abstract-** Imidazole, an important heterocycle, having biological and therapeutic properties prompted various medicinal chemists to develop various synthetic methods to synthesize a large number of novel derivatives used in chemotherapy. This fascinating group of heterocyclic compounds has interesting biological activities such as antimicrobial, anticancer, antiviral, analgesic, anti-inflammatory, anthelmintic, anticonvulsant, antiulcer, and anti-allergic activity. Various bis-imidazole derivatives introduced a great contribution not only in the field of synthetic organic chemistry but also in the field of medicinal chemistry and chemotherapy. This article aims to review the work reported in the synthesis and chemistry of bis-imidazole derivatives during past years.

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## I. INTRODUCTION

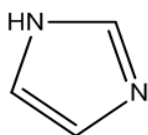
Imidazole 1, a histidine 2 component, is five membered heterocyclic compound containing nitrogen atoms in 1 and 3 positions. It has fascinating biological activities such as antibacterial [1, 2, 3], anticancer [4], antiviral [5], anti depressant [3, 6]. Bis-imidazole is an important moiety in therapeutic

chemistry due to its versatile synthesis methods and biological activities.

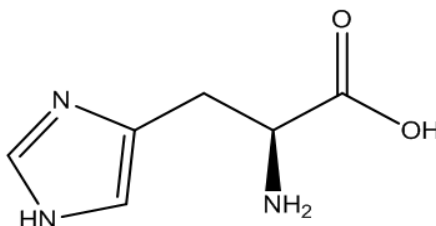
Imidazole is a five membered aromatic heterocyclic compound, categorized as diazole having two nitrogen atoms. Many important drugs contain imidazole ring, such as antifungal drugs, Nitroimidazole [7-12], and Purine 3 which is the most widely occurring nitrogen-containing heterocycle in nature consist of imidazole fused to pyrimidine [3]

Due to the significant biological activity of bis-imidazole derivatives and the rise of practical, safe, and environmentally friendly procedures for the synthesis of bis-imidazoles, [13-15] organic chemists become interested in the development of novel bis-imidazole derivatives.

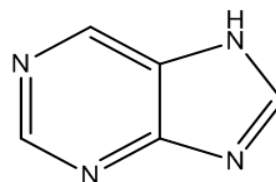
The synthesis of bis-imidazole derivatives in literature does not fit with their increasing biological importance. Herein we try to put in front of organic synthesis community the few novel bis-imidazole derivatives that were synthesized and found a unique application in the last years.



1



2



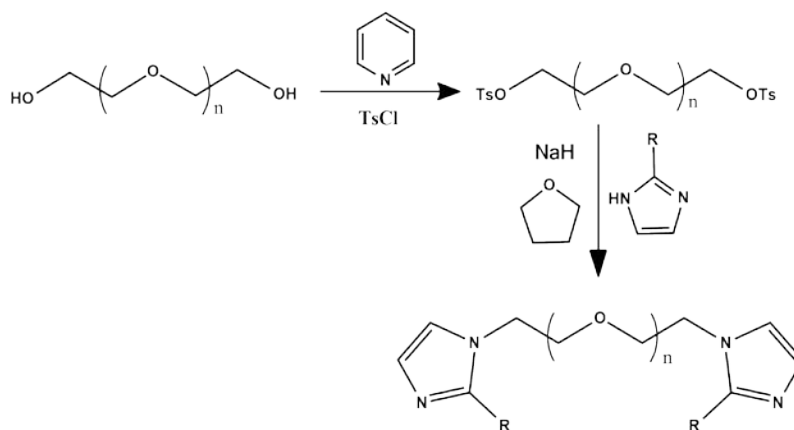
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## II. GENERAL METHODS OF SYNTHESIS

Imidazole moieties and substituted imidazoles can be formed through various methods. Different synthesis methods include Debus synthesis, Radiszewski synthesis, dehydrogenation of imidazolines, imidazole from alpha halo ketones, Wallach synthesis, from aminonitrile and aldehyde and Marckwald synthesis [12, 16]. Herein we introduce the various methods applied for the synthesis of bis-imidazole derivatives.

## III. SYNTHESIS OF BIS (IMIDAZOLE)

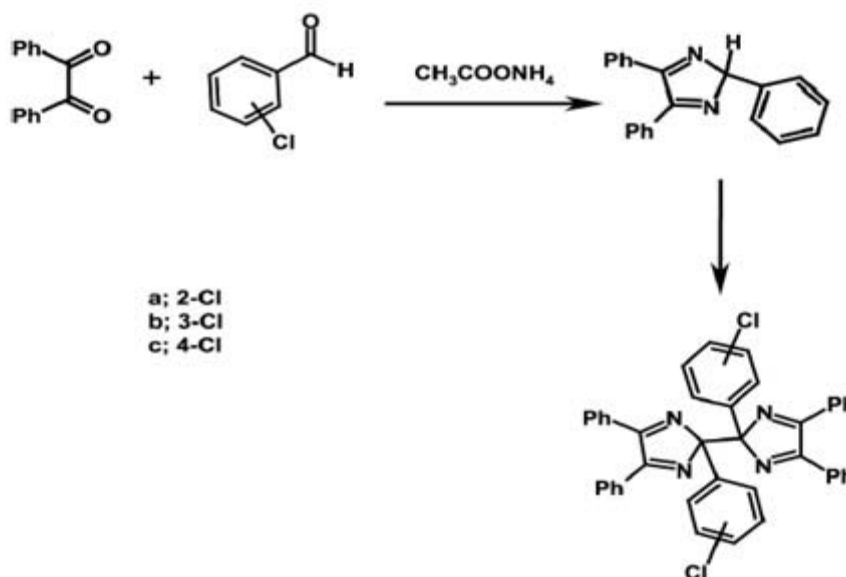
Zhou *et al.* proposed a simple, convenient and efficient method for synthesis of ether bis-imidazoles and their derivatives [17] from oligo-ethyleneglycols, imidazole, and 2-methylimidazole. The synthetic approach is shown in scheme (1).



Scheme (1)

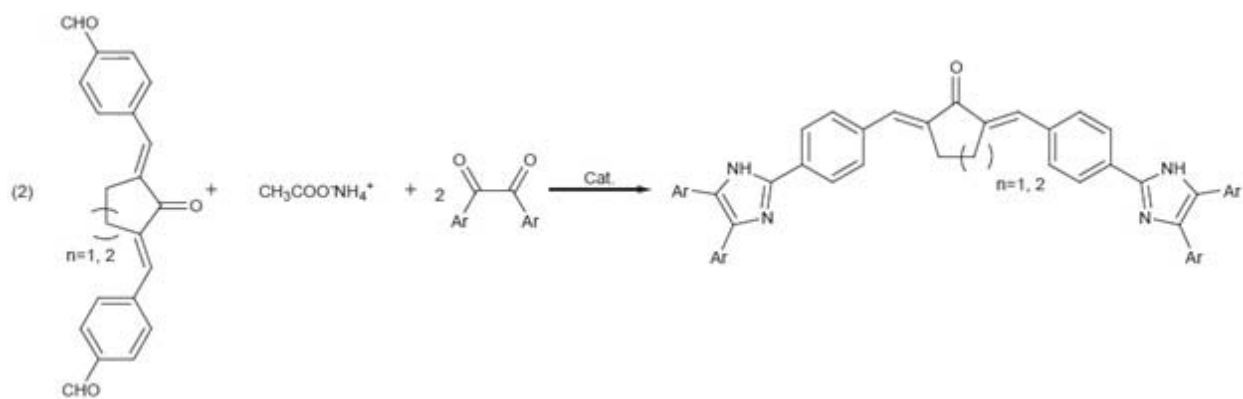
Bis-imidazole also synthesized by Asiri *et al.* [18]. He and his coworkers synthesized firstly imidazole from benzyl, ammonium acetate, and one of the three isomers of chlorobenzaldehyde. Then new bis-

imidazoles was synthesized by dissolving the imidazole derivative and cooling in ice bath, then adding potassium ferricyanide to the cooled imidazole, the synthesis route is shown in scheme (2).



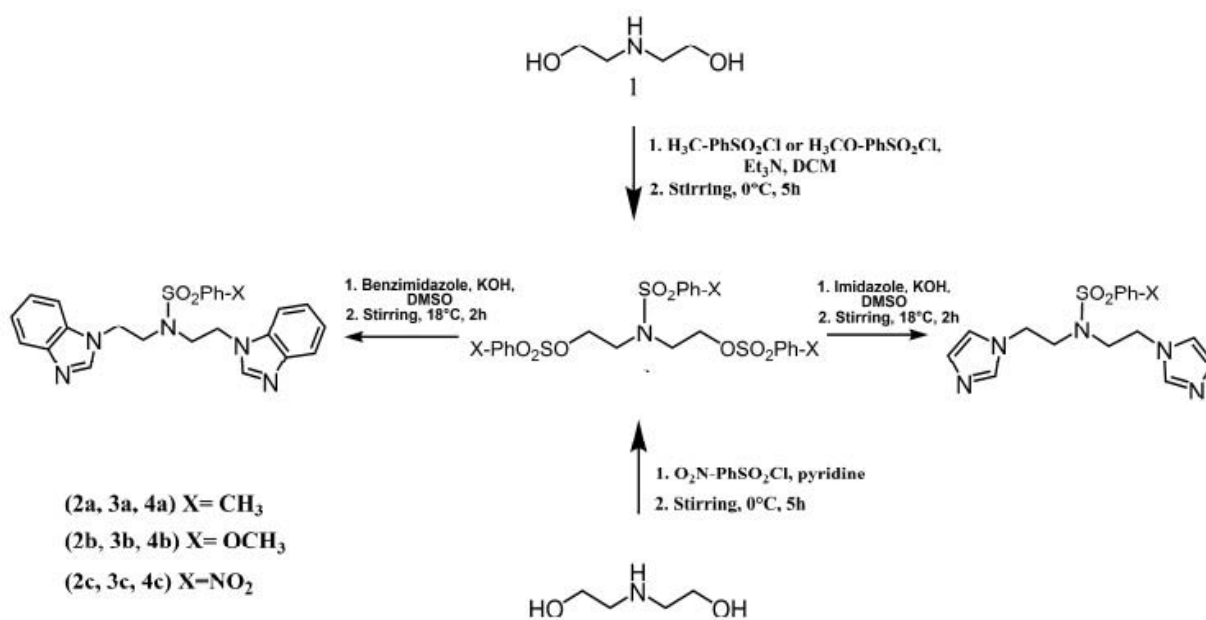
Scheme (2)

Ghazvini *et al.* reported an efficient one-pot synthesis for some novel bis imidazole from 4,4'-(2-oxocycloalkane-1,3-diylidene) bis(methan-1-yl-1-ylidene) dibenzaldehydes derivatives with benzyl and ammonium acetate [15]. The reaction conditions were optimised by changing the type and amount of catalyst, and adjusting reaction temperature. The key advantages of this procedure are environmentally friendly conditions and good yield of products. The synthetic pathway is shown in scheme (3).



Scheme (3)

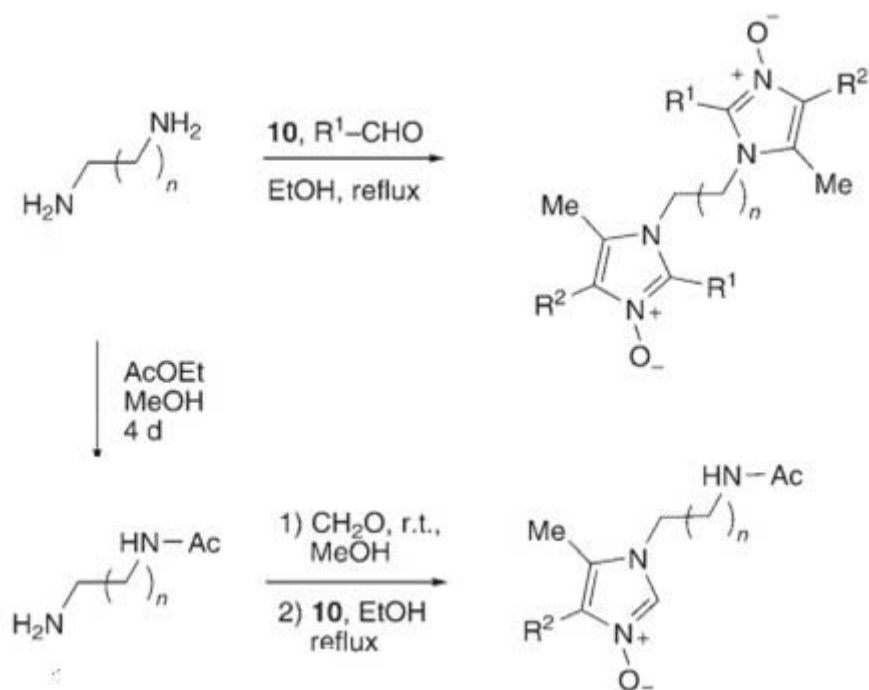
Nassir *et al.* synthesized some novel bisimidazole sulfonamide compounds [19]. The bisimidazole sulfonamides compounds were obtained by treating imidazole with tris-(4-substituted benzensulfonate)-diethanolamine under basic conditions to form the corresponding bis imidazole as shown in scheme (4).



Scheme (4)

Jasinski *et al.* afford an interesting method for synthesis of some novel bis-imidazoles [20]. The synthesis procedure involved the formation of bis-imidazole oxide compounds that were converted finally to bis-imidazole. The bis-imidazole oxide derivatives synthesis step was carried out according to scheme (5).

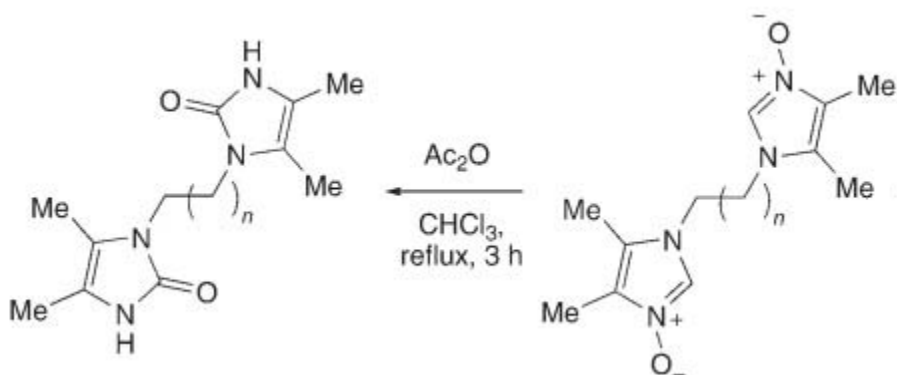




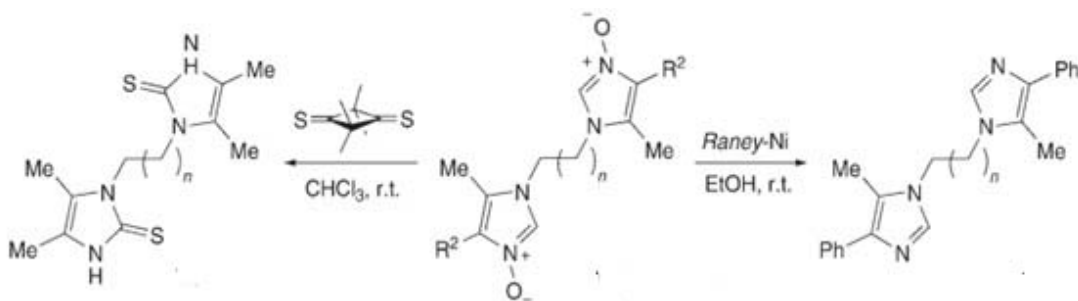
Scheme (5)

The prepared bis[imidazole 3-oxides] was isomerized with  $\text{Ac}_2\text{O}$  to give the bis[2H-imidazol-2-one] as shown in scheme (6). Finally bis[imidazole 3-oxides] derivatives was converted to 2H-imidazole-2- thiones

and bis-imidazole derivatives using 2,2,4,4-tetramethylcyclobutane-1,3-dithione and Raney-Ni respectively according to scheme (7).



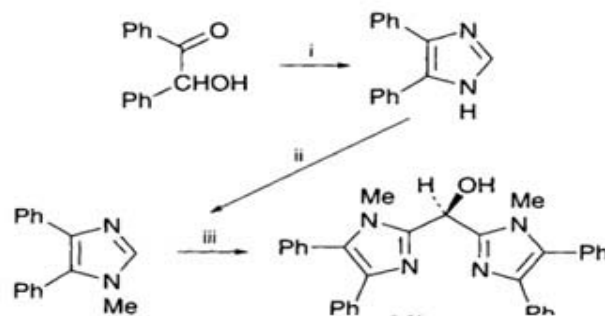
Scheme (6)



Scheme (7)

Bhalla *et al.* studied the coordination ability bis-imidazole, to achieve their goal they prepared Bis(1-methyl-4,5-diphenylimidaz-2-oyl) (benzyloxy) methane

ligand [21, 22] using the conditions followed in scheme (8).



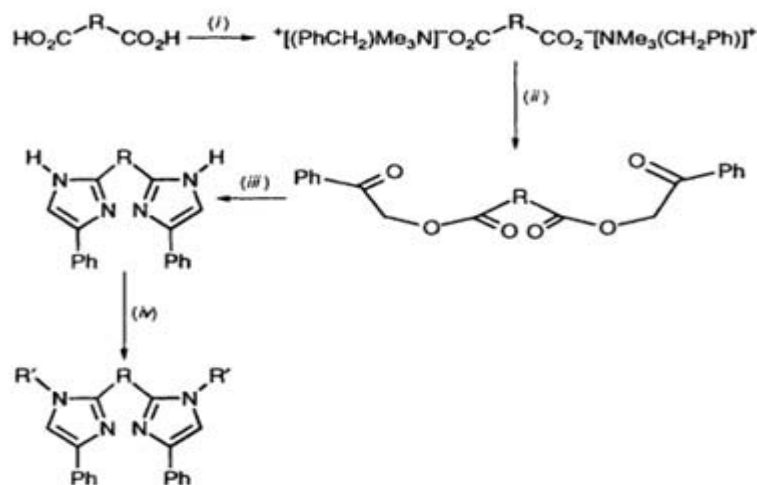
Synthesis of bis(1-methyl-4,5-diphenylimidaz-2-oyl)carbinol,

BimOH. Reagents and conditions: i,  $\text{H}_2\text{NCHO}$ ; ii, NaH then MeI, thf; iii, BuLi then 0.5  $\text{HCO}_2\text{Et}$ , thf,  $-78^\circ\text{C}$ .

Scheme (8)

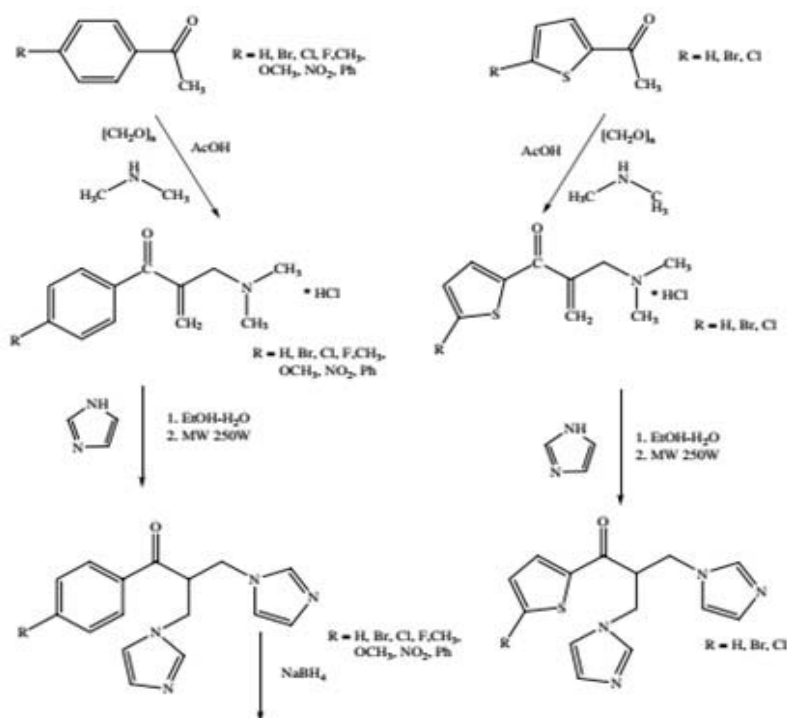
Due to presence of donor nitrogen atoms, imidazole heterocycle is of great interest in the coordination chemistry. To be used as a proligands, Higgs *et al.* reported the synthesis of new sterically hindered bis(imidazole). The authors followed a multi step synthesis as shown in scheme (9), which lead to the formation of 1,2-bis-(4-phenylimidazol-2-yl)ethane [23]. The first step involves dissolving succinic acid in MeOH and neutralised to pH 7.5 using  $[\text{NMe}_2(\text{CH}_2\text{Ph})][\text{OMe}]$  (40% solution in MeOH). In the

following step Formamide was then added to the reaction mixture and the solution stirred for 5 min before bromoacetophenone was added. In the third step ammonium acetate is introduced to the reaction liquor and refluxed for 48 hours. Here we have prepared Dimethylbis(4-phenylimidazol-2-yl)Imethan. The authors extended their work to another final step which involves the use of sodium hydride to achieve deprotonation of the obtained bis(imidazole).



Scheme (9)

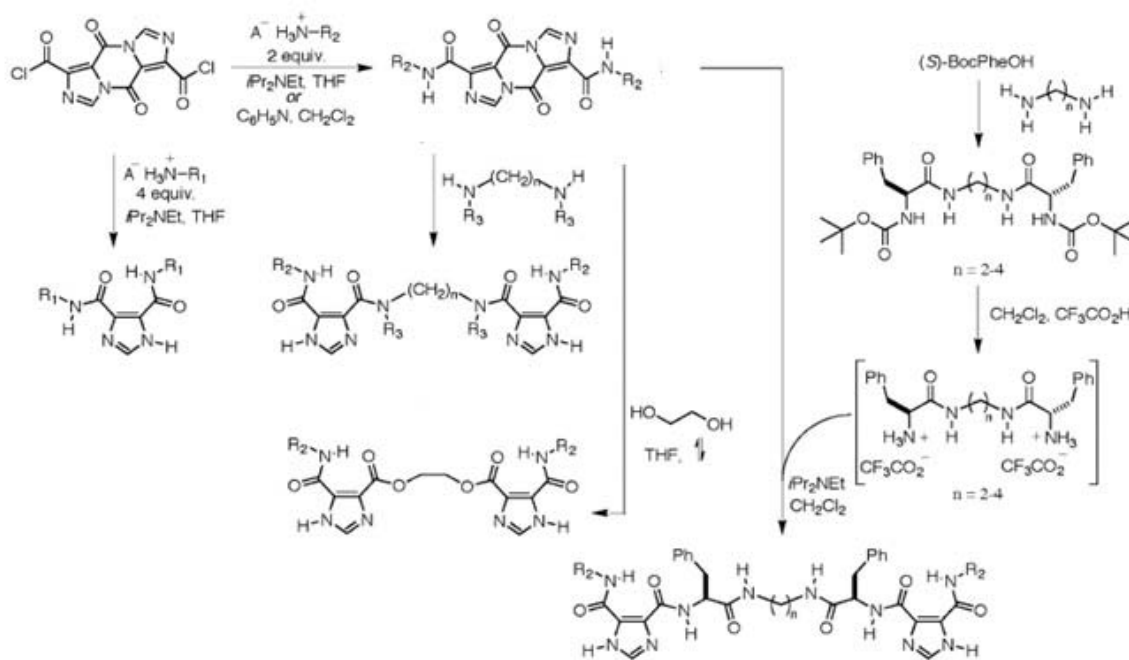
New bis-imidazole derivatives have been synthesized by Zampieri *et al.* [24]. The synthesis was achieved using via a Michael type reaction by nucleophilic attack of imidazole to 1-Aryl-2-[(dialkylamino)methyl]-propenone hydrochlorides or and 2-[(dimethylamino)methyl]-1-(thiophene-2-yl)-propenones under microwave (MW) irradiation of the reagent mixture in  $\text{EtOH-H}_2\text{O}$  at room temperature.



Scheme (10)

Wiznycia *et al.* prepared a series of symmetric bis(imidazole-4,5-dicarboxamides) with amino acid esters and a variety of linker groups [25]. The bis-imidazole derivatives in this study were obtained by

reacting a symmetric pyrazine derivative bearing reactive acid chloride and acyl imidazole functionalities with 2 equiv of an amino acid salt along with a base scheme (11).



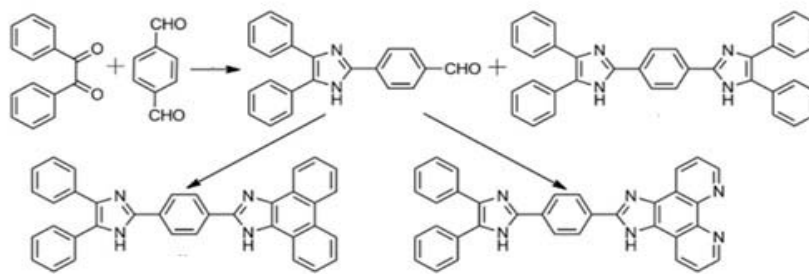
Scheme (11)

Zhang *et al.* reported a scheme involves the synthetic procedure for the synthesis of bis-imidazole compounds according to previous studies [26, 27, 28]

as illustrated in scheme (12). In this study, the authors synthesized one symmetrical structure called 1,4-bis(4,5-diphenyl-1H-imidazol-2-yl)benzene and two

asymmetrical structures, namely 2-(4-(4,5-diphenyl-1*H*-imidazol-2-yl)phenyl)-1*H*-phenanthro (9,10-*d*) imidazole and 2-(4-(4,5-diphenyl-1*H*-imidazol-2-yl)phenyl)-1*H*-imidazo(4,5-*f*)(1,10)phenanthroline. The synthetic route

of bis-imidazole compounds begins with terephthalaldehyde, benzyl, and ammonium acetate in presence of glacial acetic acid.



Scheme (12)

#### IV. CONCLUSION

Bis-imidazoles are an important heterocycles having great biological activities and used as an important precursor for the synthesis of various other bioactive compounds. Several synthetic routes and methods have been introduced to achieve the synthesis of these compounds. This review highlighted the strategies and synthetic routes for the bis-imidazole synthesis.

#### Conflict of Interest

The authors confirm that this article content has no conflict of interest.

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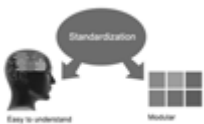






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Global Journals Incorporation (USA) is accredited by Open Association of Research Society, U.S.A (OARS) and in turn, affiliates research institutions as “Institutional Fellow of Open Association of Research Society” (IFOARS).

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The IFOARS institution is entitled to form a Board comprised of one Chairperson and three to five board members preferably from different streams. The Board will be recognized as “Institutional Board of Open Association of Research Society”-(IBOARS).

*The Institute will be entitled to following benefits:*



The IBOARS can initially review research papers of their institute and recommend them to publish with respective journal of Global Journals. It can also review the papers of other institutions after obtaining our consent. The second review will be done by peer reviewer of Global Journals Incorporation (USA). The Board is at liberty to appoint a peer reviewer with the approval of chairperson after consulting us.

The author fees of such paper may be waived off up to 40%.

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The IBOARS can organize symposium/seminar/conference in their country on behalf of Global Journals Incorporation (USA)-OARS (USA). The terms and conditions can be discussed separately.

The Board can also play vital role by exploring and giving valuable suggestions regarding the Standards of “Open Association of Research Society, U.S.A (OARS)” so that proper amendment can take place for the benefit of entire research community. We shall provide details of particular standard only on receipt of request from the Board.



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After nomination of your institution as “Institutional Fellow” and constantly functioning successfully for one year, we can consider giving recognition to your institute to function as Regional/Zonal office on our behalf.

The board can also take up the additional allied activities for betterment after our consultation.

### **The following entitlements are applicable to individual Fellows:**

Open Association of Research Society, U.S.A (OARS) By-laws states that an individual Fellow may use the designations as applicable, or the corresponding initials. The Credentials of individual Fellow and Associate designations signify that the individual has gained knowledge of the fundamental concepts. One is magnanimous and proficient in an expertise course covering the professional code of conduct, and follows recognized standards of practice.



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- The professional accredited with Fellow honor, is entitled to various benefits viz. name, fame, honor, regular flow of income, secured bright future, social status etc.



- In addition to above, if one is single author, then entitled to 40% discount on publishing research paper and can get 10% discount if one is co-author or main author among group of authors.
- The Fellow can organize symposium/seminar/conference on behalf of Global Journals Incorporation (USA) and he/she can also attend the same organized by other institutes on behalf of Global Journals.
- The Fellow can become member of Editorial Board Member after completing 3yrs.
- The Fellow can earn 60% of sales proceeds from the sale of reference/review books/literature/publishing of research paper.
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- • This individual has learned the basic methods of applying those concepts and techniques to common challenging situations. This individual has further demonstrated an in-depth understanding of the application of suitable techniques to a particular area of research practice.

## Note :

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- In future, if the board feels the necessity to change any board member, the same can be done with the consent of the chairperson along with anyone board member without our approval.
- In case, the chairperson needs to be replaced then consent of 2/3rd board members are required and they are also required to jointly pass the resolution copy of which should be sent to us. In such case, it will be compulsory to obtain our approval before replacement.
- In case of “Difference of Opinion [if any]” among the Board members, our decision will be final and binding to everyone.

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# PREFERRED AUTHOR GUIDELINES

**We accept the manuscript submissions in any standard (generic) format.**

We typeset manuscripts using advanced typesetting tools like Adobe In Design, CorelDraw, TeXnicCenter, and TeXStudio. We usually recommend authors submit their research using any standard format they are comfortable with, and let Global Journals do the rest.

Alternatively, you can download our basic template from <https://globaljournals.org/Template.zip>

Authors should submit their complete paper/article, including text illustrations, graphics, conclusions, artwork, and tables. Authors who are not able to submit manuscript using the form above can email the manuscript department at [submit@globaljournals.org](mailto:submit@globaljournals.org) or get in touch with [chiefeditor@globaljournals.org](mailto:chiefeditor@globaljournals.org) if they wish to send the abstract before submission.

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2. Authors must accept the privacy policy, terms, and conditions of Global Journals.
3. Ensure corresponding author's email address and postal address are accurate and reachable.
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- Writings
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- Printed material
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The following is the official style and template developed for publication of a research paper. Authors are not required to follow this style during the submission of the paper. It is just for reference purposes.



### ***Manuscript Style Instruction (Optional)***

- Microsoft Word Document Setting Instructions.
- Font type of all text should be Swis721 Lt BT.
- Page size: 8.27" x 11", left margin: 0.65, right margin: 0.65, bottom margin: 0.75.
- Paper title should be in one column of font size 24.
- Author name in font size of 11 in one column.
- Abstract: font size 9 with the word "Abstract" in bold italics.
- Main text: font size 10 with two justified columns.
- Two columns with equal column width of 3.38 and spacing of 0.2.
- First character must be three lines drop-capped.
- The paragraph before spacing of 1 pt and after of 0 pt.
- Line spacing of 1 pt.
- Large images must be in one column.
- The names of first main headings (Heading 1) must be in Roman font, capital letters, and font size of 10.
- The names of second main headings (Heading 2) must not include numbers and must be in italics with a font size of 10.

### ***Structure and Format of Manuscript***

The recommended size of an original research paper is under 15,000 words and review papers under 7,000 words. Research articles should be less than 10,000 words. Research papers are usually longer than review papers. Review papers are reports of significant research (typically less than 7,000 words, including tables, figures, and references)

A research paper must include:

- a) A title which should be relevant to the theme of the paper.
- b) A summary, known as an abstract (less than 150 words), containing the major results and conclusions.
- c) Up to 10 keywords that precisely identify the paper's subject, purpose, and focus.
- d) An introduction, giving fundamental background objectives.
- e) Resources and techniques with sufficient complete experimental details (wherever possible by reference) to permit repetition, sources of information must be given, and numerical methods must be specified by reference.
- f) Results which should be presented concisely by well-designed tables and figures.
- g) Suitable statistical data should also be given.
- h) All data must have been gathered with attention to numerical detail in the planning stage.

Design has been recognized to be essential to experiments for a considerable time, and the editor has decided that any paper that appears not to have adequate numerical treatments of the data will be returned unrefereed.

- i) Discussion should cover implications and consequences and not just recapitulate the results; conclusions should also be summarized.
- j) There should be brief acknowledgments.
- k) There ought to be references in the conventional format. Global Journals recommends APA format.

Authors should carefully consider the preparation of papers to ensure that they communicate effectively. Papers are much more likely to be accepted if they are carefully designed and laid out, contain few or no errors, are summarizing, and follow instructions. They will also be published with much fewer delays than those that require much technical and editorial correction.

The Editorial Board reserves the right to make literary corrections and suggestions to improve brevity.



## FORMAT STRUCTURE

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

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### **Title**

The title page must carry an informative title that reflects the content, a running title (less than 45 characters together with spaces), names of the authors and co-authors, and the place(s) where the work was carried out.

### **Author details**

The full postal address of any related author(s) must be specified.

### **Abstract**

The abstract is the foundation of the research paper. It should be clear and concise and must contain the objective of the paper and inferences drawn. It is advised to not include big mathematical equations or complicated jargon.

Many researchers searching for information online will use search engines such as Google, Yahoo or others. By optimizing your paper for search engines, you will amplify the chance of someone finding it. In turn, this will make it more likely to be viewed and cited in further works. Global Journals has compiled these guidelines to facilitate you to maximize the web-friendliness of the most public part of your paper.

### **Keywords**

A major lynchpin of research work for the writing of research papers is the keyword search, which one will employ to find both library and internet resources. Up to eleven keywords or very brief phrases have to be given to help data retrieval, mining, and indexing.

One must be persistent and creative in using keywords. An effective keyword search requires a strategy: planning of a list of possible keywords and phrases to try.

Choice of the main keywords is the first tool of writing a research paper. Research paper writing is an art. Keyword search should be as strategic as possible.

One should start brainstorming lists of potential keywords before even beginning searching. Think about the most important concepts related to research work. Ask, "What words would a source have to include to be truly valuable in a research paper?" Then consider synonyms for the important words.

It may take the discovery of only one important paper to steer in the right keyword direction because, in most databases, the keywords under which a research paper is abstracted are listed with the paper.

### **Numerical Methods**

Numerical methods used should be transparent and, where appropriate, supported by references.

### **Abbreviations**

Authors must list all the abbreviations used in the paper at the end of the paper or in a separate table before using them.

### **Formulas and equations**

Authors are advised to submit any mathematical equation using either MathJax, KaTeX, or LaTeX, or in a very high-quality image.

### **Tables, Figures, and Figure Legends**

Tables: Tables should be cautiously designed, uncrowned, and include only essential data. Each must have an Arabic number, e.g., Table 4, a self-explanatory caption, and be on a separate sheet. Authors must submit tables in an editable format and not as images. References to these tables (if any) must be mentioned accurately.



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Figures are supposed to be submitted as separate files. Always include a citation in the text for each figure using Arabic numbers, e.g., Fig. 4. Artwork must be submitted online in vector electronic form or by emailing it.

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Although low-quality images are sufficient for review purposes, print publication requires high-quality images to prevent the final product being blurred or fuzzy. Submit (possibly by e-mail) EPS (line art) or TIFF (halftone/ photographs) files only. MS PowerPoint and Word Graphics are unsuitable for printed pictures. Avoid using pixel-oriented software. Scans (TIFF only) should have a resolution of at least 350 dpi (halftone) or 700 to 1100 dpi (line drawings). Please give the data for figures in black and white or submit a Color Work Agreement form. EPS files must be saved with fonts embedded (and with a TIFF preview, if possible).

For scanned images, the scanning resolution at final image size ought to be as follows to ensure good reproduction: line art: >650 dpi; halftones (including gel photographs): >350 dpi; figures containing both halftone and line images: >650 dpi.

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## TIPS FOR WRITING A GOOD QUALITY SCIENCE FRONTIER RESEARCH PAPER

Techniques for writing a good quality Science Frontier Research paper:

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

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

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

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

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

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

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

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

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

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

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

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

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





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

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

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

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

## INFORMAL GUIDELINES OF RESEARCH PAPER WRITING

### Key points to remember:

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

### Final points:

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

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

### The discussion section:

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

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

### General style:

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

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



### *Mistakes to avoid:*

- Insertion of a title at the foot of a page with subsequent text on the next page.
- Separating a table, chart, or figure—confine each to a single page.
- Submitting a manuscript with pages out of sequence.
- In every section of your document, use standard writing style, including articles ("a" and "the").
- Keep paying attention to the topic of the paper.
- Use paragraphs to split each significant point (excluding the abstract).
- Align the primary line of each section.
- Present your points in sound order.
- Use present tense to report well-accepted matters.
- Use past tense to describe specific results.
- Do not use familiar wording; don't address the reviewer directly. Don't use slang or superlatives.
- Avoid use of extra pictures—include only those figures essential to presenting results.

### **Title page:**

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

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

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

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

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

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

### **Approach:**

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

### **Introduction:**

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



*The following approach can create a valuable beginning:*

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

#### **Approach:**

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

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

#### **Procedures (methods and materials):**

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

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

#### **Materials:**

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

#### **Methods:**

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

#### **Approach:**

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

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

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

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



**Results:**

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

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

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

**Content:**

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

**What to stay away from:**

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

**Approach:**

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

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

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

**Figures and tables:**

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

**Discussion:**

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

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

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



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

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

#### **Approach:**

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

Describe generally acknowledged facts and main beliefs in present tense.

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| <b>Methods and Procedures</b> | 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          |
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| <b>References</b>             | Complete and correct format, well organized  | Beside the point, Incomplete  | Wrong format and structuring                                   |





# INDEX

---

---

## **A**

Adhesion · 9, 11, 14, 16

---

## **C**

Chemotherapy · 22

---

## **E**

Eluting · 12

Endosulfan · 1

---

## **F**

Fascinating · 22

---

## **G**

Gaiaswat · 18

---

## **I**

Imidazole · 22, 23, 24, 25, 26, 27, 28

---

## **P**

Penetrates · 11

---

## **V**

Vanillin · 19



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