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(Volume No. 10, Issue No. 1, January - April 2024)

# Contents

Sr. No.	Title / Authors Name	Pg. No.
1	Preparation & Characterization of Schiff's Base Ligand 2-Hydroxy-1-Naphthaldehyde Alkanesulfonylhydrazone (Nafash) – Manish Kaushik, Atul Kumar	01 - 05
2	Effect Of Sodium Chloride On Excess Enthalpy Of N- Butylamine - Water System – Dr. A. Elango., Dr. B. Karunanithi	06 - 13
3	Effects Of Temperature On Nigerian Waxy Crude Oil – Igwilo K.C., Anawe P. A.L., Orodu O.D., Okechukwu L T., Okolie T.A.	14 - 18
4	Synthesis, Characterization And Biochemical Studies Of Some Novel Glutamic Acid Derivatives Of RNA & DNA Bases – Manish Chaudhari, Sachin Patel, Krupa Vegda	19 - 28
5	Improving The Dissolved Oxygen Levels In Waste Water Using Oxygen Concentrator – Anita Kumari, Mohammad Salique, Nabila Rumane, Rohan Bholla, Siddharth Bhawnani	29 - 37

# Preparation & Characterization of Schiff's Base Ligand 2-Hydroxy-1-Naphthaldehyde Alkanesulfonylhydrazone (Nafash)

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# ABSTRACT

Three new ligands 2-hydroxy-1-naphthaldehyde propanesulfonylhydrazone( $SB^1$ ), 2 - hydroxy -1-naphthaldehyde butanesulfonylhydrazone( $SB^2$ ), 2-hydroxy -1 -naphthaldehyde pentanesulfonylhydrazone ( $SB^3$ ) were prepared. The ligands were characterized on the basis of physical properties, elemental analysis data, infrared and nuclear magnetic resonance spectroscopy. The prepared ligand shows good antibacterial activity.

Keywords: Schiff's bases, antibacterial activities, coordination chemistry

## **1. INTRODUCTION**

A Schiff base is a functional group that contains a <u>carbon-nitrogen double bond</u> with the nitrogen atom connected to an <u>aryl</u> or <u>alkyl</u> group but not <u>hydrogen</u>. Schiff bases are of the general formula  $R_1R_2C=N-R_3$ , where  $R_3$  is an aryl or alkyl group that makes the Schiff base a stable <u>imine</u>. Monodentate Schiff's bases are less known to form stable complexes, probably due to the insufficient basic strength of the imino nitrogen of the C=N group. Polydentate Schiff bases with phenolic OH or nitrogen or sulphur of the ring, suitably near to the imino nitrogen, may stabilize the metal-nitrogen bond through the formation of chelate rings. Schiff bases which are unaided by those donors but contain two **azomethine**(check spelling) nitrogen atoms, in spite of their facile ligating capabilities due to easy chances of chelation, have also been used in the CO displacement reactions of group VI metal carbonyls[1].

There is growing pharmaceutical and chemical interest in compounds containing the sulfonylhydrazene moiety[2-10]. Numerous compounds containing a sulfonamide group or a hydrazine residue, or their combination in one molecule, show cytostatic and antibacterial activity [11,12]. Sulfonamide drugs are among the most widely used chemotherapeutic agents with a large spectrum of activity [13]. Cytostatic

activity of sulfonamide derivatives attracts much attention in the last years [14–16]. Studies on their mechanism of action have revealed an important role of enzyme inhibition. Among the anticancer sulfonamides there are inhibitors of carbonic anhydrases [15–18], of cyclooxygenase-2 [19, 20], and of topoisomerases [21, 22]. Sulfonylhydrazones combine two pharmacophoric fragments, sulfonamide and hydrazine, and some of their representatives have exhibited strong antileukemic activity [23, 24].



2-hydroxy-1-naphthaldehyde alkane sulfonyl hydrazone (SB)

 $(SB^1; R=C_3H_7), (SB^2; R=C_4H_9), (SB^3; R=C_5H_{11})$ Fig. (I)

### Experimental

All solvents were dried and degassed prior to use. Elemental analysis were carried out using a Carlo Erba 1108 by Central Drug Research Institute, Lucknow, India. FTIR spectra were recorded on sample in butane at the Intertek FTIR Lab, Mumbai, India on a Perkin-Elmer Spectrum RX1 FT spectrophotometer. <sup>1</sup>H NMR spectra were recorded in CDCl<sub>3</sub> or DSMO on a 500 MHz Bruker FT-NMR at Tata Institute of Fundamental Research, Mumbai, India. UV irradiations were performed with a medium-pressure 400 W mercury lamp in quartz bulb.

The solvents, 2-hydroxy-1-naphthaldehyde, propanesulfonyl chloride, butanesulfonyl chloride, pentanesulfonyl chloride, hydrazine hydrate and silica gel were purchased from E. Merck and Aldrich. These reagents were used as supplied.

#### **Prepapation of Ligands**

2-hydroxy-1-naphthaldehyde propanesulfonylhydrazone (SB<sup>1</sup>), 2-hydroxy-1-naphthaldehyde butanesulfonylhydrazone (SB<sup>2</sup>) and 2-hydroxy-1-naphthaldehyde pentanesulfonylhydrazone (SB<sup>3</sup>) were prepared by the literature method [10]. For the preparation of 2-hydroxy-1-naphthaldehyde propanesulfonylhydrazone (SB<sup>1</sup>) a solution of 4.55 g (10mmol) of propanesulfonylhydrazine in 5ml of water was mixed with hot solution of 2.06 g (12mmol) of 2-hydroxy-1-naphthaldehyde in 10ml of

ethanol and attired for 1h. Upon cooling the obtained crystalline precipitates were filtered, washed with ethanol-ether, recrystallized from benzene-ethanol (7:1 mixture) and dried in vacuo over  $P_2O_5$ . Yield of recrystallized product is 62%.

**Solubility:** Freely soluble in DMF and DMSO and insoluble in methanol, nitrobenzene, dioxane and water.

Other ligands 2-hydroxy-1-naphthaldehyde butanesulfonylhydrazone  $(SB^2)$  and 2-hydroxy-1-naphthaldehyde pentanesulfonylhydrazone  $(SB^3)$  were prepared similarly by replacing propanesulfonylhydrazine with butane and pentane substituted sulfonylhydrazone.



#### **Results and Discussion**

The physical and analytical data for the prepared ligands is given in Table 1, which supports the formation of expected ligands, which was confirmed by IR and NMR spectroscopic data (Table 2). The evidence about Schiff's base stable imine group is the intense C=N stretching vibration, found at 1620cm<sup>-1</sup> in ligands. The other IR bands were observed for the  $v_{as}(SO_2)$ ,  $v_{sym}(SO_2)$ , and v (NH) stretching vibrations indicating that SO<sub>2</sub> and NH groups in ligands. In the proton NMR spectra of ligands, NH proton was found in ligand at 11.08 ppm and OH protons at 11.78.

Ligand	Molecular Formula	Found (Calculated), %				
Ligand		С	н	Ν	s	Mol. Wt
(5D1)	CUONS	56.79	5.22	9.16	10.50	290.86
(38-)	C <sub>14</sub> H <sub>16</sub> O <sub>3</sub> N <sub>2</sub> S	(57.52)	(5.52)	(9.58)	(10.97)	(292.35)

(5D2)	C U O N C	58.00	5.79	8.92	10.19	104.62
(SB2)	$C_{15}H_{18}O_{3}N_{2}S$	(58.80)	(5.92)	(9.14)	(10.47)	(306.38)
(SB <sup>3</sup> )	CUONS	59.09	5.97	8.44	9.81	318.98
	C <sub>16</sub> H <sub>20</sub> O <sub>3</sub> N <sub>2</sub> S	(59.98)	(6.29)	(8.74)	(10.01)	(320.41)

Table 2. Selected IR bands (cm<sup>-1</sup>) and <sup>1</sup>H-NMR (ppm) data of ligands

Ligand	Name of Ligands	Abbrevia tion	Vsym (N-H)	Vsym (C=N)	Vsym. (C-O)	<sup>1</sup> H-NMR (in CDCl <sub>3</sub> ) δ ppm
a	2-hydroxy-1- naphthaldehyde propanesulfonylhyd razone	(SB1)	3221	1624	1245	1.16 (s, 3H, CH <sub>3</sub> -CH <sub>2</sub> -CH <sub>2</sub> ), 3.11 (s, 2H,CH <sub>2</sub> -CH <sub>2</sub> ), 4.23 (s, 2H, CH <sub>2</sub> -S), 7.21, 7.36, 7.54, 7.86, 7.93, 8.52 (m, 6H, C <sub>12</sub> H <sub>6</sub> ), 8.61 (s, 1H, HC=N), 10.85 (s, 1H, NH), 11.78 (s, 1H, OH)
b	2-hydroxy-1- naphthaldehyde <u>butanesulfonylhydr</u> <u>azone</u>	(SB <sup>2</sup> )	3220	1623	1247	1.16 (s, 3H, CH <sub>3</sub> -CH <sub>2</sub> -CH <sub>2</sub> -CH <sub>2</sub> ), 3.13 (s, 2H,CH <sub>2</sub> -CH <sub>2</sub> -CH <sub>2</sub> ), 4.22 (s, 2H, CH <sub>2</sub> -CH <sub>2</sub> ), 4.65 (s, 2H, CH <sub>2</sub> -S), 7.21, 7.36, 7.54, 7.86, 7.93, 8.52 (m, 6H, C <sub>12</sub> H <sub>6</sub> ), 8.61 (s, 1H, HC=N), 10.85 (s, 1H, NH), 11.78 (s, 1H, OH)
с	2-hydroxy-1- naphthaldehyde pentanesulfonylhyd razone	(SB <sup>3</sup> )	3222	1622	1245	1.16 (s, 3H, CH <sub>3</sub> -CH <sub>2</sub> -CH <sub>2</sub> -CH <sub>2</sub> -CH <sub>2</sub> -CH <sub>2</sub> ), 3.11 (s, 2H,CH <sub>2</sub> -CH <sub>2</sub> -CH <sub>2</sub> -CH <sub>2</sub> ), 4.23 (s, 2H, CH <sub>2</sub> -CH <sub>2</sub> -CH <sub>2</sub> ), 4.73 (s, 2H, -CH <sub>2</sub> -CH <sub>2</sub> ), 4.91(s, 2H, CH <sub>2</sub> -S) 7.21, 7.36, 7.54, 7.86, 7.93, 8.52 (m, 6H, C <sub>12</sub> H <sub>6</sub> ), 8.61 (s, 1H, HC=N), 10.85 (s, 1H, NH), 11.78 (s, 1H, OH)

#### Antibacterial Activity

All the synthesized ligands were screened for their antibacterial activity by using agar diffusion method [25] against *S. aureus*, *B. subtile* gram positive and *E. coli*, *S. paratyphi* gram negative bacteria in nutrient agar medium. Ciprofloxacin was used as standard drug for comparison.

These schiff's Bases show antibacterial activity against *E. coli, S. aureus, B. subtile* and *S. paratyphi*. It is found that 2-hydroxy-1-naphthaldehyde propanesulfonylhydrazones( $SB^1$ ) studied here follow such a decomposition scheme in the cell, and diazoalkane intermediates further could alkylate the nucleophilic centres of biologically important macromolecules and in particular DNA. 2-hydroxy-1-naphthaldehyde butanesulfonylhydrazones( $SB^2$ ) and 2-hydroxy-1-naphthaldehyde

pentanesulfonylhydrazones(SB<sup>3</sup>) shows moderate activity.

#### Conclusions

 $SB^1$ ,  $SB^2$  &  $SB^3$  ligands were prepared by thermal reaction of 2-hydroxynapthaldehyde and alkanesulfonylhydraone. The results show that alkanesulfonylhydrazones and its ligand derivatives exert a moderate inhibitory effect on different bacterial activity, thus confirming our previous data about the cytotoxic activity of these compounds.

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# Effect Of Sodium Chloride On Excess Enthalpy Of N- Butylamine -

# Water System

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# ABSTRACT

Separation of liquid mixtures by the extractive distillation technique employing an inorganic or organic salt instead of the liquid separating agents, that are used conventionally, has the potential to yield higher separation efficiency with attendant advantages like lower energy requirements and lesser cost of equipments. The salt added plays an effective role by changing the Vapour-liquid equilibrium relationship in the view of the liquid phase turning into a solution of an electrolyte whose degree of dissociation becomes a function dependant on the relative proportions of the two volatile components. In this investigation the effect of sodium chloride on excess enthalpy of binary miscible Butylamine-Water system was studied with weight percentage 5%, 10% and 15% of sodium chloride, it is found that the increase in the exothermic peak value of the system with increase in the salt concentration is due to the fact that the added salt favors association by acting as a polarizing medium.

Keywords: Extractive Distillation, Exothermic, Model Parameter, Vapour liquid Equilibrium, Excess Enthalpy

## **1. INTRODUCTION**

In the process of separation of liquid mixtures by the extractive and azeotropic distillation methods, the addition of a third component is a common technique. Such third components are added to alter the relative volatility or to alter or to eliminate azeotropes for the purpose of facilitating distillation. Recent studies have suggested that solid agents may also be used as separating agents in a dissolved form. Such three component systems consisting two liquids and one salt, have been encountered in various industrial processes. In many cases, distillation affords the most economical means of separating the liquid components. In the design of distillation equipment, the vapour liquid equilibrium relationship between the two components is of prime importance. Hence, any factors, affecting the equilibrium relationship, will in turn affect the separation efficiency. Here it may be noted that the presence of salt is one such factor which affects the equilibrium relationship .Although the exact nature of the action of the

salt, dissolved in a liquid, is not certain, it is understood that the presence of salt alters the relative volatility of the liquid by lowering its vapour pressure. The extent of the change in volatility depends on the solubility of the salt in the liquid. In extractive distillation, the inorganic salt is added as an extractive agent to modify the relative volatility of the binary mixture. The selective effect that the salt can have on the volatilities of the two liquid components and hence on the composition of the equilibrium vapor comes about primarily through the effect exerted by salt ions and/or molecules on the structure of the liquid phase. In this investigation the effect of sodium chloride on excess enthalpy of binary miscible Butylamine-Water system was studied with weight percentage 5%, 10% and 15% of sodium chloride.

#### LITERATURE REVIEW

Rajendran et al <sup>[23,38]</sup> studied the effect of inorganic salts (Calcium chloride, Sodium chloride and Zinc Chloride) in vapor liquid equilibria and heat of mixing of the methanol-Ethyl acetate system. They found that the addition of calcium chloride brought about a significant enhancement in the heat of mixing while the salts sodium chloride and zinc chloride decrease the lateral shift. Rajendran et al [24,39] studied the effect of sodium chloride, calcium chloride and zinc chloride on excess enthalpy for systems methanol-water, methanol-benzene, pyridine-water. They found that for methanol-water system the salt sodium chloride decrease the exothermic value with increase in salt concentration while the calcium chloride found to shift the excess enthalpy to the endothermic side. They further found for the system methanol-benzene the salt calcium chloride and zinc chloride increased the endothermic excess enthalpy. Rajendran et al<sup>[21]</sup>Studied the effect of Sodium Chloride, Calcium Chloride and Zinc Chloride at the concentration 5wt% to 30 wt% on Liquid-Liquid Equilibria of the ternary system Ethyl acetate – 2. Proponal-Water and Vapor Liquid Equilibria of its constituent binary system (ie) Ethyl acetate- 2-propanol, 2-Propanol-Water and ethyl acetate-water. The system 2-propanol-water had both positive and negative values of the excess enthalpy with exothermic behavior in propanal lean region. All the salts which were preferentially soluble in water were found to decrease the exothermic value steadily as their concentration was increased. In the case of 2.proponal -ethyl acetate system, the salt sodium chloride and zinc chloride had brought about a small change in the magnitude of heat of mixing, while the addition of calcium chloride had resulted in a decrease in the value of heat of mixing in ester rich region and increase in ester lean region. A shift in the peak value was observed when calcium chloride salt was added due to the possible formation of alcohol –salt complex. Rajendran et al <sup>[22,40,43]</sup> studied the effect of sodium chloride, calcium chloride and zinc chloride on heat of mixing of 2, proponal-Benzene system. They found that the salt sodium chloride and zinc chloride brought about an enhancement in heat of mixing values and the salt calcium found to produce the opposite effect. Frice et al<sup>[7]</sup> studied the excess enthalpies of methanol, ethanol, 1-propanol, 2-propanol and aqueous solution

of NaCl, KCl values of excess enthalpy were measured using flow calorimeter at various Temperatures mixtures which are alcohols. The temperature at which the investigation was carried out are 285.65, 298.15, 308.15 and 323.15 K. Those with ethanol and propanol were additionally investigated at 338.15 K. The concentration of the salt water component was varied between 0 and 10% in water for 2-proponal + water system. That excess enthalpy of mixture of aqueous solution containing 7.5 weight percentage. NaCl at 308.15 K was reduced about one third compared to that of salt free system. For the mixture of ethanol, 1-prpoponol or 2- proponol with water the addition of KCl causes reduction of the exothermic effect by 13-16%.

#### **MATERIALS USED**

The solvents used in this investigation are obtained from E-Merck India Ltd. and also from British Drug House Labs India. The hydrocarbon are assayed by gas chromatography and exceeds 99.5% purity and some of the solvents are further purified by fractional distillation. Anhydrous grade salts are used and all salts are dried for about 24 hrs before use. The physical properties of solvents and salts are given in Tables 1 and 2.

	Boilin	g Point C	Densit	y at 30°C	Refractic Index at 30°C		
Components	This work	<u>Timmermans</u> et al(1962)	This work	Timmermans et al (1962	This work	Timmermans et al(1962)	
Butylamine	77.8	78.1	0.738	0.735	1.390	1.389	
Water	100	100.10	0.9971	1.0102	1.3325	1.3330	

Table 1 Physical Properties of the solvents used

#### Table 2 Physical properties of salt

Salt	Purity	Molecular weight	Specific gravity	Melting point °C	Solubility in water at 30°C (gms/100cc)
Sodium Chloride	99.98	82.03	1.528	324	119

## **Experimental Calorimeter**

The calorimeter is designed to operate isothermally so as to allow the excess enthalpy at a given temperature to be determined for the entire composition range of a system in two experimental runs. A temperature probe is used as the temperature sensing device and the isothermal conditions are easily maintained within  $\pm 0.005^{\circ}$ C. The set temperature is readily reproduced within  $\pm 0.002^{\circ}$ C. The calorimeter contains vapour space but and source of error due to condensation and vaporization

are very negligible, operating procedure of the calorimeter are described in Rajendran et al <sup>[24,41,42]</sup>

#### **RESULTS AND DISCUSSION**

linear plots of  $H^{E}/x_{1}x_{2}$  vs  $x_{1}$  and  $x_{1}x_{2}/H^{E}$  vs  $x_{1}$  for salt free butylamine-water are shown in Figures 1 and 2 and the effect of NaCl on Butyl amine-water system is shown in Figure 3 and in the Table 3. The exothermic peak value of the system for 5wt% NaCl is -1816 J/mol obtained at 0.416 mol fraction of Butyl amine and for 10 wt% NaCl the exothermic peak value is - 2268 J/mol obtained at 0.3827 mol fraction Butyl amine and for 15 wt% NaCl the exothermic peak value is -2406 J/mol obtained at 0.476 mol fraction Butyl amine. The increase in the exothermic peak value of the system with increase in the salt concentration is due to the fact that the added salt favors association by acting as a polarizing medium.

No	Salt	5	5 wt % <u>NaCl</u>		10 wt % NaCl		1	15 wt % <u>NaCl</u>		
<b>x</b> <sub>1</sub>	HE	<b>x</b> 1	x <sub>3</sub>	HE	<b>x</b> <sub>1</sub>	<b>X</b> 3	HE	x <sub>1</sub>	<b>X</b> 3	HE
0.11	-900	0.11	0.0327	-960	0.109	0.0682	-1196	0.141	0.098	-1265
0.2	-1240	0.2	0.0252	-1459	0.196	0.0518	-1805	0.3	0.0589	-2114
0.25	-1400	0.251	0.0142	-1641	0.258	0.0429	-2075	0.38	0.0434	-2327
0.33	-1600	0.333	0.0106	-1801	0.3039	0.0376	-2196	0.476	0.0326	-2406
0.42	-1600	0.416	0.00782	-1816	0.3827	0.0296	-2268	0.568	0.0241	-2314
0.54	-1400	0.537	0.00512	-1629	0.479	0.0222	-2158	0.682	0.0159	-1986
0.65	-1000	0.649	0.0033	-1298	0.575	0.0613	-1881	0.763	0.0103	-1621
0.78	-600	0.7575	0.00205	-895	0.689	0.0107	-1407	0.854	0.00642	-1091
					0.777	0.00701	-986	0.952	0.0022	-389

Table 3 Effect of NaCl on Excess Enthalpy of Butylamine(1)+Water(2) system



 $Figure \ 1 \quad H^E/x_1x_2 \ vs \ x_1 \ for \ System \ Butylamine(1) + Water(2) \ at \ 30^{o}C$ 





Figure 3 Effect of <u>NaCl</u> on Excess <u>Enthapy</u> of <u>Butylamine(1)</u>+Water(2) System at 30°C

## CORRELATION OF EXPERIMENTAL EXCESS ENTHALPY DATA

The experimental data obtained in this investigation are fitted in the Redlich-Kister equation to obtain the parameters and to find the average deviation from the experimental results. The equation is given by

$$H^{E}/x_{i}(1-x_{j}) = \sum a_{i}(2x_{j}-1)^{i}$$

where  $a_0, a_1, a_2, ..., a_n$  are Redlich equation constants and  $\sigma$  is average deviation.

Salt	Wt% of salt	a <sub>0</sub>	a1	a2	<b>a</b> 3	a4	σ
	-	-5526	2731	28.35	3.28	46.31	1.23
NaCl	5	-6834	3813	16.85	-21.36	-45.74	1.3
NaCI	10	-8439	4955	1.713	2.532	-2.08	1.56
	15	-9591	1191	7.75	-6.27	-16.49	1.75

Table 4 Parameters of <u>Redlich-Kister</u> equation

### CONCLUSION

The exothermic excess enthalpy of system N- Butylamine–Water increases with increase in concentration of sodium chloride. The results of this experiment-oriented investigation suggested that the salt in the dissolved form could bring about significant changes in the behavior of liquid mixtures which in turn are likely to have considerable influence on the economics of distillation and liquid extraction techniques.

#### List of symbols

a	-	constants of typical polynomial
f(x)	-	General form a typical fitting function
$\mathrm{H}^{\mathrm{E}}$	-	Excess enthalpy
i	-	Subscript of polynomial
m	-	Number of data points
n	-	Order of polynomial
$\mathbf{X}_{1}$	-	Mol fraction of component 1
<b>X</b> <sub>2</sub>	-	Mol fraction of component 2
X <sub>3</sub>	-	Weight fraction of salt
$\mathbf{X}_{i}$	-	Independent variable (typically mole fraction)
Σ	-	Summation symbol

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# Effects Of Temperature On Nigerian Waxy Crude Oil

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# <u>ABSTRACT</u>

This technical paper evaluates the effects of temperature on Nigerian waxy crude oil. The laboratory measurements were carried out on wax appearance temperature, using the precipitation method. The transient state measurement, the degree of viscosity reduction and the yield stress of the given crude oil were also determined. It is noted that the crude oil sample has no transient behavior since the shear stress shows time independent behavior. It can be observed that the yield stress which was required to start the flow decreased as the temperature increases. The results of the study shows that the percent wax deposited decreased as temperature increases. Also the percent Degree of Viscosity Reduction increased from 20OC to 60OC. Also, the Nigerian waxy crude oil has no transient behavior and the yield stress decreased as the temperature increases.

Keywords: Temperature, Nigerian waxy crude oil, Transient state, Yield stress, Degree of viscosity reduction.

#### **1. INTRODUCTION**

The severity of paraffin deposition depends predominantly on the composition (wax content) of the crude oil, the cloud-and pour-points, the rate of evolution of gas, well depth, formation temperature, the ambient and operating temperatures, pressure drop, previous shear history, pipe roughness and production practices. Cloud-point is the temperature at which a haze forms in clear oil which is being cooled. For a crude oil, it is the temperature at which paraffin particles begin to precipitate out of solution. Generally, depending on the flow regime, wax deposition begins to occur on any surface referred to as cold spot whose temperature is below this critical cloud-point and that of the bulk oil. The pour- point is defined as the temperature below which the oil ceases to flow (pour). The pour-point is usually 10°F-20°F lower than the cloud-point. In the case of asphaltene deposition, the severity depends mainly on the composition of the crude oil, operating pressure and temperature and the enhanced oil recovery methods. It is known in both cases that the severity of the problem increases with the age of

the oil field. Thus the term waxy crude is used to embrace both kinds of organic deposition. Of the two kinds of deposition, paraffin deposition is the most widespread and more easily handled while asphaltene deposition is much more difficult to treat because of the complex depositional relationship between the crude oil composition, pressure and temperature.

Wax is a commonly occurring component of crude oil. It is generally characterized as consisting of large n-paraffins that are solid at room temperature when isolated. Yet, they are soluble in the crude oil mixture at elevated temperatures. Waxy crude oils are crude oils with relatively high amount of wax suspended in them (1). It is found that n-paraffin dissolved in organic solvents display a sharp transition in gel strength at the pour point, whereas by addition of isoparaffins, the buildup in gel strength as a function of temperature is much more gradual, because increasing isoparaffin fraction facilitates the formation of amorphous wax solids (2).

At high temperatures, waxes are in the molten state, and crude oils normally behave like Newtonian fluids (3). If waxy crude is allowed to cool to the temperatures below the Wax Appearance Temperature (WAT), wax will precipitate, agglomerate and entrap the liquid oil into its structure, and the crude will become a two-phase dispersion with wax solid particles dispersed in the liquid hydrocarbons. Precipitation of wax significantly increases crude viscosity and will gradually change the flow properties of the crude from Newtonian to non- Newtonian behavior. The crude begins to show non-Newtonian flow behavior at a temperature called the abnormal point which is generally a few degree Celsius below the WAT. Further cooling of the crude enhances the gel formation.

#### MATERIALS AND METHODS

A crude oil sample from the Niger-Delta basin is collected. The characteristics of the crude oil sample are presented in Table1. Initially, the crude oil is homogenized by shaking it for an hour to ensure that the crude oil's physical properties are the same anywhere the sample is taken for the measurements. The measurements are taken at room temperature.

SAMPLE	А
SPECIFIC GRAVITY	0.91
API	23.99
POUR POINT	- 19°C
CLOUD POINT	- 13°C
ТҮРЕ	INTERMEDIATE CRUDE

## Table 1: Characteristics of the Crude Oil Sample.

Laboratory measurements were carried out on wax appearance temperature, followed by the transient test was also conducted in order to know the behavior of the crude oil sample with respect to time.

We also carried out a study to know the % Degree of Viscosity Reduction, that is, the degree of viscosity reduction (%DVR) was introduced and calculated using the equation below and the result shown in table 4.

 $DVR\% = (\Pi r - \Pi c) * 100 / \Pi r$ 

Where  $\Pi$ ris the reference viscosity at 50s-1 shear rate and 20°C, and  $\Pi$ c is the corresponding viscosity at 50s-1 shear rate and corresponding temperature. Yield stress was also determined at different temperatures as shown in table 5.

### **RESULTS AND DISCUSSION**

Four tables of data results which include wax appearance temperature, % degree of viscosity reduction, transient measurements and yield stress were then generated and their results then discussed. Table 2: Wax precipitated at different temperatures

Temperature	Weight of residue	Weight of	Wax precipitated
(°C)	(g)	precipitate (g)	(%)
-10	1.29	0.83	45.60
0	0.96	0.50	27.47
10	0.79	0.33	18.13
20	0.72	0.26	14.29
30	0.65	0.19	10.44
40	0.57	0.11	6.04
50	0.52	0.06	3.30
60	0.46	0.00	0.00

Table 3: DVR % of crude oil versus temperature

Temperature, °C	DVR %
20	0
30	61
50	78
60	81

25°C 45°C		45°C		65°C	
τ <sub>.</sub> Pa	t, s	τ, Pa	t, s	τ, Pa	t, s
53.7	30	20.5	30	9.20	30
53.7	60	20.5	60	9.20	60
53.7	100	20.5	100	9.20	100
53.7	140	20.5	140	9.20	140
53.7	170	20.5	170	9.20	170
53.4	200	20.5	200	9.20	200
53.4	230	19.9	230	9.20	230
53.4	270	19.9	270	9.20	270
53.4	300	19.9	300	8.70	300

Table 4: Transient Measurements at Constant 600 rpm

Table 5: Yield Stress of the Crude Oil Sample at different temperatures

Yield Stress, Pa	Temperature °C
2.04	20
0.67	30
0.62	50
0.47	60

From tables 2 to 5 above, they following discussions were made. % Wax precipitated decreased as temperature increases.

Also, the % Degree of Viscosity Reduction (% DVR) over the temperature range of 20°C to 60°C occur. A significant increase in %DVR from 0 to 81 was noticed as the temperature increases from 20°C to 60°C. This can be attributed to strong effect of temperature on the viscosity of heavy components of the crude. The second reason is due to the effect of high temperature on the chemical structure of heavy components in the crude oil phase, and hence reducing the oil viscosity.

It is noted that the crude oil sample has no transient behavior since the shear stress shows time independent behavior. It can be observed that the yield stress which is required to start the flow decreased as the temperature increases.

#### CONCLUSIONS

• Wax precipitates out of the waxy crude oil at temperatures below its wax appearance temperature (WAT). And as the temperature decreases below the wax appearance temperature, the amount of wax

precipitated increases.

- The yield stress required to start the flow of the crude oil sample decreased with increase in temperature.
- The crude oil sample did not show any transient behavior, hence there is no time dependent.

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# Synthesis, Characterization And Biochemical Studies Of Some Novel Glutamic Acid Derivatives Of RNA & DNA Bases

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# ABSTRACT

Amino acid derivatives of RNA & DNA bases constituent an important class of heterocyclic compounds. These organic syntheses organized in solvent phase with the help of microwave irradiation. Nucleobases having considerable heterocyclic and amino acids are also having high physiological activity. With this multi-valued objective, the newly synthesized compound highly intensified. Many of them observed antifungal, antibacterial, anti-cancer, anti-inflammatory activities. The structures of newly synthesized compounds have been determined with the help of elemental analysis like IR, 13C NMR, and Mass spectral data.

Keywords: RNA & DNA base, Glutamic Acid, Antibacterial activity, Antifungal activity.

#### **1. INTRODUCTION**

In human body, proteins are very important and very big complex molecules. From which type of units is made up, it is called amino acid. In heterocyclic compound, amino acids and its derivatives of RNA &DNA bases are very important. It gives many biological activities like antimicrobial (antibacterial & antifungal), anti- inflammatory, anticancer. It supports the central nervous system and act as a neurotransmitter and play a very important role in the treatment of depression, schizophrenia, anxiety and mood connected problems and also gives protection from heart attacks.Necleobase are also having very much biological importance. So it attract to synthesized Glutamic Acid derivatives of RNA & DNA base.

#### 2. MATERIALS AND METHOD

The derivatives of amino acid have been made by reacting with different RNA & DNA base. All the chemicals are pure and granted so we used without further purification. We used Glutamic Acid,

Adenine, Guanine, Thymine, Uracil, Ethanol, and Distilledwater.

General procedure for synthesis of various RNA &DNA base & amino acid derivative Glut amic Acid and RNA & DNA base were weighed equally in respect to the moles. The properly weighed compounds were thoroughly mixed well using distilled water. Then mixture is transferred into RBF (250 ml). Put the RBF into microwave oven. Set the microwave at full microwave radiation (900 W) as per reaction time and start the microwave oven. After the completion of reaction the RBF is taken from the oven very carefully. Then the reaction mixture is transferred into evaporating dish and evaporates the mixture and collects the dry product.Recrystallized from hot water. When we were used guanine, the reaction is take place in ethanol on behalf of water.

#### (1) Product M<sub>4</sub>A (Glutamic Acid + Adenine)



 $M_4A$ 

#### (2) Product M<sub>4</sub>C (Glutamic Acid + Guanine)



 $M_4C$ 

#### (3) Product M<sub>4</sub>D (Glutamic Acid + Thymine)



#### (4) Product M<sub>4</sub>E (Glutamic Acid + Uracil)



 $M_4E$ 

#### **3. SPECTRA CHARACTERIZATION**

#### (1) Compound M<sub>4</sub>A:

#### Infrared Spectra Feature (cm<sup>-1</sup>):

1594.45: -NH<sub>2</sub> NH<sub>2</sub> bend

1149.05, 1173.50, 1212.76, 1256.37: -C-N Stretch

1669.37:- C=N Stretch

### 3049.11:- OH (Carboxylic Group)

1750.58:- C=O Stretch (Ketone)

1558.71:-C=C Aromatic Stretch

701.19, 778.91: -C-H Aromatic out of plane bends

1413.59, 1450.25:-CH<sub>3</sub>,CH<sub>2</sub>

### (2) Compound M<sub>4</sub>C:

### Infrared Spectra Features (cm<sup>-1</sup>):

1558.71, 1508.21: -NH<sub>2</sub> NH<sub>2</sub> bend

[11149.05, 1173.50, 1212.76, 1256.37]:C-N Stretch

1670.46, 1697.25:- C=N Stretch

3115.27, 3315.58:- OH(Carboxylic Group)

1770.69:-C=O Stretch (Ketone)

1558.71:-C=C Aromatic Stretch

## <sup>13</sup>C spectral Feature (ppm):

40.09, 39.88, 39.60, 39.47, 39.26, 39.05, 38.84:- R<sub>2</sub>-CH<sub>2</sub>, R<sub>3</sub>-CH, C-N

155.65:- C=O

152.36:-C=C

#### Mass spectral Features (ppm):

112.1:- Peak is observed due to  $C_{5}H_{7}NO_{2}$ . This is Glutamic Acid peak

69.1:-Base peak is observed due to  $C_4H_8N$  This is Glutamic Acid peak

701.19, 778.91:-C-H Aromatic out of plane bends

1370.37, 1416.45, 1458.26,1473.85: - CH<sub>3</sub>, Ch<sub>2</sub>

#### <sup>13</sup>C spectral Features (ppm):

[40.09,39.88, 39.68, 39.47, 39.26, 39.05, 38.84]:- R<sub>2</sub>-CH<sub>2</sub>, R<sub>3</sub>-CH, - C-N

#### Mass spectral Features (ppm):

135.1:-Peak is observed due to  $C_5H_3N_4O$ . This is Guanine peak.

112.1:- Base peak is observed due to C<sub>3</sub>H<sub>7</sub>NO<sub>2</sub>. This is Glutamic acid Peak

#### (3) Compound $M_4D$ :

#### **Infrared Spectra Features (cm<sup>-1</sup>):** 1506.46, 1556.11: -Nhbends

2760.65, 3030.73: -OH (Carboxylic Group) 1748:-C=O Stretch (Ketone)

1589.58:-C=C Aromatic Stretch

709.82, 756.77, 805.77, 862.62, 941.59: С-Н

Aromatic out of plane bends

1350.92, 1416.22:- CH<sub>3</sub>CH<sub>2</sub>

[1050.60, 1086.46, 1125.22, 1150.31, 1206.30, 1350.92]:- C-N Stretch

1642.74, 1668.37: -C=N Stretch

<sup>13</sup>C spectral Features (ppm): 11.17:- R-CH<sub>3</sub>

(4) Compound M<sub>4</sub>E:

#### Infrared Spectra Features (cm<sup>-1</sup>):

1558.33: -NH bend

3649.13:-OH(Carboxylic Group) 1746:-C=O Stretch (Ketone) 1506.66:-C=C Aromatic Stretch

69.1:- Base peak is observed due to  $C_4H_8N$ . This is Glutamic acid Peak

40.07, 39.07, 39.66, 39.05, 39.24, 38.03,

38.02:- R<sub>2</sub>-CH<sub>2</sub>, R<sub>3</sub>-CH, C-N

164.89:-R-COOH, C=O

137.69, 107.65:-C=C

151.46:- C in Aromatic ring

#### Mass spectral Features (ppm):

197.2:- Peak is observed due to  $C_8H_{10}N_3O_3$ 

126.1:-Peak is observed due to  $C_5H_5N_3O$ . This is Thymine peak 69.1:-Base peak is observed due to  $C_4H_8N_3$ . This is Glutamic Acid peak 112.1:- Peak is observed due to  $C_5H_6N_2O$ .

711.86, 806.99, 846.74, 910.87, 944.49:С-Н Aromatic out of plane bend 1418.99, 1457.45:- CH<sub>3</sub> CH<sub>2</sub>

[1051.73, 1083.24, 1149.71, 1256.95, 1312.62]:- C-N Stretch

1650.53, 1683.38:- C=N Stretch

## <sup>13</sup>C spectral Features (ppm):

40.04, 39.83, 39.62, 39.42, 39.21, 38.00, 38.79:- R<sub>2</sub>-CH<sub>2</sub>, R<sub>3</sub>-CH, C-N

164.32:-R-COOH,C=O

100.18:- C=C

151.49, 147.17:- C in Aromatic ring

#### Mass spectral Features (ppm):

149.1:- Peak is observed due to  $C_6H_5N_3O_2$ .

112.1:-Base peak is observed due to  $C_4H_4N_3O$ . This is Uracil peak

84.1:-Peak is observed due to C<sub>4</sub>H<sub>5</sub>NO. This is Glutamic Acid peak

Sr.	Compound	M.P	Nitrogen	Rule		Compound	Base	Unsaturation
No	Name		Rule	Of		Formula	Formula	Index (U)
				13			$C_n H_{n+r}$	
				n	r			
1	M <sub>4</sub> A	182°C	Yes	20	4	$C_{10}H_{12}N_6O_3$	C <sub>20</sub> H <sub>24</sub>	15
2	$M_4C$	210°C	Yes	21	7	$C_{10}H_{12}N_6O_4$	C <sub>21</sub> H <sub>28</sub>	15
3	M <sub>4</sub> D	218°C	Yes	19	8	$C_{10}H_{13}N_3O_5$	C <sub>19</sub> H <sub>27</sub>	13
4	$M_4E$	290°C	Yes	26	3	C <sub>9</sub> H <sub>11</sub> N <sub>3</sub> O <sub>5</sub>	C <sub>26</sub> H <sub>29</sub>	19

Table 1: Various Derivatives of Glutamic acid

## 4. ANTIMICROBIAL ACTIVITY

In antimicrobial activity, antibacterial activity is pulls down the growth of bacteria and gives protection against bacterial infection. Many drugs like antibiotics and other chemicals performed antibacterial activity. The radical of antibiotics is diffused by the agar, when we added antibiotics in agar cup. If the organism is having minimum concentration, we observed clear zone of retardation. If the zone is large then the MIC is vast. The size of zone is depends on the concentration of compound.

In conical flask culture is melted and cooled at 50  $^{\circ}$ C. Add nutrient agar and put for solidify. In sterile agar plate add different antibiotics. Put plate in the incubate at 37  $^{\circ}$ C for 24 hours. After 24 hours zone is observed in the plate. Largest zones are considered the smallest minimum inhibitory concentration (MIC) of antibiotic for that bacteria or fungi.

In antifungal activity different pathogenic organisms are used. The agar cup method was used for antifungal activity. This activity was performed same as antibacterial activity.

We have used the **Broth Dilution Method** to evaluate the antibacterial activity.

The main advantage of the **'Broth Dilution Method'** for MIC determination lies in the fact that it can readily be converted to determine the MIC as well.

1. Serial dilutions were prepared in primary and secondary screening.

- 2. The control tube containing no antibiotic is immediately sub cultured [before inoculation] by spreading a loopful evenly over a quarter of plate of medium suitable for the growth of the test organism and put for incubation at 37 °C OVERNIGHT. The tubes are then incubated overnight.
- 3. In antimicrobial the MIC is lowest concentration (mg/L) that inhibits the growth of the microorganisms.
- 4. In the ICU, infection is caused by the pathogens with higher MIC.
- 5. In critically ill patients, regular MIC is required to decrease with the using antibiotics.

Bacteria	Zone inhibition					
	Gentamycin	n <u>Chloramphenlcol</u> Ciprofloxacin <u>Norfloxa</u>				
		Ampicillin				
E coli	0.05	100	50	25	10	
P.Areuginosa	1	0	50	25	10	
S.Aureus	0.25	250	50	50	10	
S.Pyogenus	0.5	100	50	50	10	

## Table 2: Antibacterial Activity of Standarddrug

#### Table 3: Activity Bacterial of Compounds

Bacteria	Zone inhibition				
	M <sub>4</sub> A	M <sub>4</sub> D	$M_4E$		
E coli	500	100	100		
P.Areuginosa	50	62.5	200		
S.Aureus	25	250	500		
S.Pyogenus	62.5	250	500		

#### Table 4: Antifungal Activity of Standarddrug

Fungi	Zone inhibition				
	Nystatin	Greseofulvin			
<u>C.Albicans</u>	100	500			
<u>A.Niger</u>	100	100			
<u>A.Clavatus</u>	100	100			

Fungi	Zone inhibition				
	M <sub>4</sub> A	M <sub>4</sub> D	$M_4E$		
C.Albicans	>1000	>1000	>1000		
<u>A.Niger</u>	>1000	200	200		
<u>A.Clavatus</u>	>1000	200	500		

#### **Table 5: Antifungal Activity of Compounds**

#### **RESULT AND DISCUSSION**

According to observation Table3 Sample contain MIC range 0.001 ml to 0.005 ml constitute 0.01 mg in 10 ml solvent. The activity of  $M_4A$ ,  $M_4D$ , and  $M_4E$  extract is observed between 25 mm to 500 mm. against respective strain. At each strain lowest MIC activity observed in standard drugs minimum 0.05mm andmaximum 250 mm. This activity indicate zone of inhibition against various bacterial strain such as *E.coli, P.Areusinasa, S.Aureus and S.Pyagenls* of same dilution. The activity of standard drug was given in Table 2.

Antibacterial activity of compound M4A, M4D, and M4E are excellent as compare to the standard drug at same concentration.

According to observation Table 5 Sample contain MIC range 0.001 ml to 0.005 ml constitutemg in 10 ml solvent. The activity of M4A, M4D, and M4E extract is observed between 125 mm to 250 mm. against respective strain. At each strain lowest MIC activity observed in standard drugs minimum 100 mm andmaximum 500 mm. This activity indicate zone of inhibition against various fungal strain such as C.Albicans, A.Niger, A.Clavatus of same dilution. The activity of standard drug was given in Table 4.

Antifungal activity of compoundsM4A, M4D, and M4E are excellent as compare to the standard drug at same concentration.

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Manish Chaudhari<sup>1</sup>, Jahnavi Darji<sup>1</sup>, Dipa Dabhi<sup>1</sup>, Dhaval Bhanotar<sup>2</sup>, Dilip Gami<sup>2</sup> International Research Journal of Natural and Applied Sciences

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# Improving The Dissolved Oxygen Levels In Waste Water Using Oxygen Concentrator

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# ABSTRACT

Dissolved oxygen refers to the level of free, non-compound oxygen present in water or other liquids. It is an important parameter in assessing water quality because of its influence on the organisms living within a body of water. In the case of aeration process, dissolved oxygen is highly significant as microorganisms depend on the dissolved oxygen for their growth. The microorganisms use this dissolved oxygen in order to breakdown the organic waste present in the effluent and thus helps to reduce the Biological Oxygen Demand (BOD)/Chemical Oxygen Demand (COD) of the effluent. If the dissolved oxygen is too low, certain microorganisms might perish thus increasing the MLSS: MLVSS (Mixed liquor suspended solids: Mixed liquor suspended solids) ratio. On the other hand, if the dissolved oxygen is too high, again there is growth of unwanted micro-organisms which harm the aeration process. When ideal dissolved oxygen is maintained, maximum efficiency of organic waste breakdown in the effluent is attained. In order to serve this purpose an oxygen concentrator is being used to provide an economical source of oxygen in industrial processes. Current technology advocates the use of the Pressure Swing Adsorption (PSA) method utilizing various adsorbents, mostly zeolites to obtain upto 99% pure oxygen.

*Keywords:* Adsorbents, Aeration Process, BOD, COD, Pressure Swing Adsorption.

#### **1. INTRODUCTION**

Industrial waste water is one of the main pollution sources in the pollution of water environment. During the last century a huge amount of industrial waste water was discharged into rivers, lakes, and coastal areas. This resulted in serious pollution problems in the water environment and caused negative effects to the ecosystem and human life.

#### 2. WHY TREAT WASTEWATER?

The principal objective of wastewater treatment is generally to allow human and industrial effluents to be disposed of without danger to human health or unacceptable damage to the natural environment. We cannot allow wastewater to be disposed of in a manner dangerous to human health and lesser life forms

or damaging to the natural environment. Basic Sewage Treatment facilities reduce organic and suspended solids to limit pollution to the environment. Advancement in needs and technology has necessitated the evolving of treatment processes that remove dissolved matter and toxic substances. Currently, the advancement of scientific knowledge and moral awareness has led to a reduction of discharges through pollution prevention and recycling, with the noble goal of zero discharge of pollutants [7].

Treatment technology includes physical, biological, and chemical methods. Residual substances removed or created by treatment processes must be dealt with and reused or disposed of in a safe way. The purified water is discharged to surface water or ground water. Residuals, called sludges, may be reused by carefully controlled composting or land application [1][6].

#### **3. TYPES OF WASTES**

The types of wastes in effluent waste water are broadly divided into the following two categories:

- 1. Inorganic Waste: Waste material such as sand, salt, iron, calcium, and other mineral materials that are only slightly affected by the action of organisms. Inorganic wastes are basically chemical substances of mineral origin.
- 2. Organic Waste: Waste material that may come from animal or plant sources. Natural organic wastes generally can be consumed by bacteria and other small organisms [6].

#### 4. EFFLUENT TREATMENT PROCESS

Effluent treatment entails the treatment of all the wastewater generated in the plant. The effluent treatment plant, or ETP, is responsible for making the generated wastewater fit for reuse and also for draining of the water into municipal drains, in compliance with the standards for drainage water set by the Pollution Control Board (PCB).



Figure 1: Step-by-step ETP process

# The step-by-step ETP process is as follows:

- <u>Bar screen chamber</u>: This is where all the wastewater is first sent for pretreatment. It consists
  of multiple meshes through which the effluent water is passed. The mesh sizes are 30mm and
  3mm. This is where the big size solid waste is removed.
- 2. <u>Oil trap chamber</u>: The effluent from the bar screen is collected in this chamber. This chamber is used to separate the floating oil present in the raw effluent. Belt type motorized oil skimmer is provided for the removal of settled floating oil from the oil trap chamber and is transferred to slop oil tank by gravity. The oil and grease present in the effluent sticks to the belt provided in the skimmer and is thus removed from the effluent.
- 3. <u>Collection tank:</u> The water from the oil trap is then collected in the collection tank where some settling takes place. The pH of the collected effluent is generally in the range of 9-12
- 4. Equalization tank: Before the aeration process starts, the pH of the effluent must be brought down to 6.5-8.5 in order to facilitate proper functioning of the micro- organisms. For this purpose, the water in the equalization tank is maintained at a pH of 7-7.5, which is achieved by acid dosing. Hydrochloric acid is used for this dosing purpose.

Journal of Applied Chemistry (Volume- 10, Issue - 1 Jan-Apr 2024)

- 5. <u>Biox tank:</u> The equalized effluent water is then sent to the biox tank where a bio filling using polypropylene material is mixed with it in order to reduce the Biological Oxygen Demand (BOD) and the Chemical Oxygen Demand (COD) of the water. The BOD/COD of the effluent is brought down to nearly 50% of its original value in the Biox tank. Also, air is circulated at a relatively lower flowrate in order to facilitate microorganism growth.
- 6. <u>Aeration tank:</u> The process involves air or oxygen being introduced into the effluent combined with organisms to develop a biological floc which reduces the organic content of the sewage. This material is largely composed of saprotrophic bacteria, Spirotrichs, Peritrichs including Vorticellids and a range of other filter feeding species. This mixture is known as Mixed Liquor. The air is circulated with a relatively higher flow rate in this tank in order to increase the Dissolved Oxygen (DO), which is utilized by the introduced microorganisms to breakdown the organic waste present in the effluent. The BOD/COD is thus, brought down to 10% of the original value.
- 7. <u>Secondary Clarifiers:</u> The water from the aeration tanks is made to flow the bottom to top in this tank. The flocculated sludge is collected at the bottom and is recirculated back to the aeration tank. A part of the clarified water is sent to storage tanks as per the plant's requirement whereas the rest of the water is sent into municipal drains. All the water from the secondary clarifier is treated with 5% Sodium Meta Bi Sulphate in order to kill the Chlorine content of the treated water. The conditions of the water sent to municipal drains is as follows: pH 5-9, COD<250ppm, BOD< 30ppm, Total Dissolved Solids (TDS)< 2100ppm, Total Suspended Solids (TSS)< 100, Oil/Grease<10. The water is now totally safe to dispose of into government drains [8].</p>

#### **5. ACTIVATED SLUDGE PROCESS**

The term activated sludge is used to indicate the sludge which is obtained by settling the effluent in presence of abundant oxygen. The sludge is biologically active and it contains a great number of aerobic bacteria and other microorganisms which effectively oxidize the organic matter. After pH adjustment, the effluent is routed for two stage biological treatment for the carbonaceous organic removal. The activated sludge process essentially consists of an aeration tank with aerators, a clarifier and a sludge recirculation system. The nutrient requirement of nitrogen and phosphorous for the microbial growth will be fulfilled by the addition of urea and DAP (Diammonium phosphate) solution. The urea solution serves as the N-P source. The quantity of both the chemicals added is such as to maintain BOD:N:P

ration of 100:10:1. The neutralized effluent is received in the biox tank which is provided with aerators for COD/BOD reduction. The COD/BOD reduction at this stage is about 50% as mentioned before. The effluent ex-Biox tank is routed to the aeration tank. The BOD reduction then is about 90% the exit of the effluent from the aeration tank is routed to the clarifier where the microbial solids are separated under gravity. Solids separation is he final step in the production of a well-clarified, stable effluent, low in BOD and suspended solids and as such represents a critical link in the operation of an activated sludge treatment process. The clarifier is provided with a clarifier mechanism to remove the sludge at the bottom. The separated biomass settles in the conical shaped bottom of the clarifier. This biomass collected will be pumped via non-clog centrifugal pumps. A portion of the settled solids is recycled back to maintain the desired concentration of microorganisms in the aeration tank and excess sludge is pumped to sludge drying beds for solar drying provided in the ETP area. About 162kg of 100% bio solids is collected every day.

Aeration is generally provided to three tanks, namely, equalization, biox and aeration tank. Aeration of the effluent is done with the help of air blowers. In the case of biox and equalization tanks, air is supplied through course bubble diffusers and in aeration tank by fine membrane diffusers for homogenizing the effluent and also to keep it fresh during storage. Butterfly valve is provided to regulate the flow at the outlet of blower and to isolate the equalization tank.

## 6. SIGNIFICANCE OF DISSOLVED OXYGEN IN WATER

Dissolved oxygen refers to the level of free, non-compound oxygen present in water or other liquids. It is an important parameter in assessing water quality because of its influence on the organisms living within a body of water. A dissolved oxygen level that is too high or too low can harm aquatic life and affect water quality. Non-compound oxygen, or free oxygen ( $O_2$ ), is oxygen that is not bonded to any other element. Dissolved oxygen is the presence of these free  $O_2$  molecules within water. The bonded oxygen molecule in water ( $H_2O$ ) is in a compound and does not count toward dissolved oxygen levels. One can imagine that free oxygen molecules dissolve in water much the way salt or sugar does when it is stirred. Dissolved oxygen enters water through the air or as a plant byproduct. From the air, oxygen can slowly diffuse across the water's surface from the surrounding atmosphere, or be mixed in quickly through aeration [3].

DO can be measured in mg/l or %saturation. The level of DO in water is mainly dependent on temperature, pressure and alkalinity of water. The normal operating DO level in an aeration tank should be 1-2 mg/l (ideally 2mg/l) or 10-20% saturation. In the case of the aeration process, DO is highly

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significant as microorganisms depend on the DO for their growth. The microorganisms use this DO in order to breakdown the organic waste present in the effluent and thus helps to reduce the BOD/COD of the effluent. If the DO is too low, certain microorganisms might perish thus increasing the MLSS: MLVSS ratio. Also the growth of certain unwanted bacteria is facilitated. In this anaerobic phase, organisms also excrete certain waste products which darken the colour of the effluent. COD/BOD value too, increases significantly. On the flip side, if the DO is too high, again there is growth of unwanted microorganisms which harm the aeration process. When ideal DO is maintained, maximum efficiency of organic waste breakdown in the effluent is attained and the colour of water in the aeration tank remains golden brown. The water turns black and turbid when the DO is below the required ideal value.

# 7. METHODS TO IMPROVE THE DISSOLVED OXYGEN LEVELS IN THE ACTIVATED SLUDGE

- The air blowers cannot be used at their maximum outputs even if the DO is extremely low, because if they are, then the membrane bursts. So, bigger sized membranes should be used or membranes made of materials with higher yield stress should be used. Also, the tube type diffusers in the biox and equalization tank expand and burst if the pressure is increased beyond a particular value. Tube diffusers with larger diameters should be used to enable greater air supply. At the current point in time, not more than 2 blowers can be used, each running at not more than a third of their output capacity can be used safely.
- Oxygen concentrators should be placed before the air blowers in order to increase the oxygen quantity being supplied to the aeration process. This will drastically improve the dissolved oxygen levels in the effluent [2].
- A proper sludge removal system for the equalization tank should be in place. This must be done in order to stabilize the MLSS value of the effluent. If the sludge is removed frequently, then aeration can be provided more often to the equalization tank.
- Filters must be added between the equalization and biox tanks. This will help to bring down the MLSS value of the effluent in the aeration tank. These filters can be cleaned easily by backwash. Occasional manual cleaning might also be required.
- Partially cover the aeration tank- Partially or completely covering the tank may be an option to regulate seasonal changes. Atmospheric pressure is a seasonal factor that can affect dissolved oxygen concentrations within the tanks. Low pressure conditions, such as strong winter winds, and high pressure conditions, such as humid, stagnant By partially or completely covering the aeration tank, the plant can have more control over the efficiency of the biological processes taking place. A

retractable type geo- membrane cover would be highly beneficial in controlling temperature fluctuations and also eliminate the bad odor given off by the effluent.

Of these various methods, the most effective and efficient method to improve the dissolved oxygen level in the activated sludge is to introduce oxygen concentrators to the process

## 8. OXYGEN CONCENTRATORS

Oxygen concentrators typically use pressure swing adsorption technology and are used very widely for oxygen provision in healthcare applications, especially where liquid or pressurized oxygen is too dangerous or inconvenient, such as in homes or in portable clinics. The major technique currently employed is the Pressure Swing Adsorption (PSA) technique. Pressure swing adsorption processes rely on the fact that under high pressure, gases tend to be attracted to solid surfaces, or "adsorbed". The higher the pressure, the more gas is adsorbed; when the pressure is reduced, the gas is released, or desorbed. PSA processes can be used to separate gases in a mixture because different gases tend to be attracted to different solid surfaces more or less strongly. If a gas mixture such as air, for example, is passed under pressure through a vessel containing an adsorbent bed of zeolite that attracts Nitrogen more strongly than it does Oxygen part or all of the Nitrogen will stay in the bed, and the gascoming out of the vessel will be enriched in oxygen. When the bed reaches the end of its capacity to adsorb nitrogen, it can be regenerated by reducing the pressure, thereby releasing the adsorbed nitrogen. It is then ready for another cycle of producing oxygen enriched air [2][4].

#### 9. ADSORBENTS FOR PRESSURE SWING ADSORPTION

#### (i) Silica Gel:

Silica gel is used in a pretreatment bed to remove water vapor and impurities such as carbon dioxide and carbon monoxide before the air feed stream enters the adsorbent beds. Water strongly adheres to the cation sites within each zeolite rendering them useless and ineffective. Silica gel beds are necessary to remove water vapor from the air. Air at 100% humidity has approximately 3% of water vapor. Once the bed is saturated with water, the bed is heated with a heating coil to evaporate the water from the silica gel [5].

#### (ii) Zeolites:

Zeolites are microporous crystalline structures that govern the molecules that are adsorbed during the PSA process. The shape-selective properties of zeolites are the basis for their use in molecular adsorption. The different structures of the zeolite indicate the type of molecules that the zeolite will adsorb.



Figure 6: Zeolite structure

Zeolites have various ways of controlling adsorption. The size and shape of pores can control access into the zeolite. In another case different types of molecule enter the zeolite, but some diffuse through the channels more quickly while others are left behind and do not pass through. Cation-containing zeolites, such as silver zeolites, are extensively used in gas separation processes. These cations are indicated as the purple spheres in the figure above.

Molecules are differentiated on the basis of their electrostatic interactions with the metal ions. Zeolites can thus separate molecules based on differences of size, shape and polarity [5].

Ion exchange is another aspect of zeolites that aids in the separation process. Ion exchange involves adding metal cations to the structure of the zeolite to attract certain molecules. Calcium is the most common metal cation exchanged in zeolites, but new studies have found silver exchanged zeolites to be more effective in air separation. For zeolites to be affective, metal cations must be bound to the structure such as calcium, sodium, and in our case silver. Silver exchanged zeolites are a relatively new type of zeolite used in separation. The silver metal cation is placed in the structure of the zeolite structure as shown below. Zeolite structure types of A, X, and Y are the dominant types used in commercial use for adsorption and ion exchange [5].

#### **10. CONCLUSION**

The most promising property of oxygen concentrator for improving the dissolved oxygen level in waste water is its ability to selectively adsorb oxygen on the zeolite bed from the ambient air. This property translates into more oxygen levels per unit area. The scope of this technology is thus immense in the environmental and the health sectors. It opens up exciting avenues for a country like India where the problems are many and solutions are few.

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