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SYNTHESIS OF GUARGUM DERIVED RESIN AND IT'S APPLICATION IN INDUSTRIAL WASTE WATER MANAGEMENT

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***Abstract**: Guar amino salicylic acid (GASA) and Guar orthanilic acid (GOAA) resin were synthesized by reacting epoxy propyl ether of Guar gum with 5-amino salicylic acid and orthanilic acid. The epoxy propyl ether of guar gum was synthesized by reaction of epichlorohydrin with guar. The various physiochemical properties of the resin are examined and found that resin is stable in acidic as well as basic medium. The characterization of guar gum based resin is done by elemental analysis and FTIR (Fourier transform Infrared) studies. The removal efficiency of resin for heavy metal ions followed order:* $Cd^{2+} < Zn^{2+} < Pb^{2+}$

***Keywords:** Guar gum, 5-amino salicylic acid, orthanilic acid, epoxy propyl ether, epichlorohydrin*

1. Introduction

The major contributing factor for water pollution are waste water from different sources, intensive agriculture, industrial production, infrastructure development, untreated urban runoff and wastewater. The presence of heavy metals in wastewater has been increasing with the growth of industry and human activities, e.g. plating and electroplating industry, batteries, pesticides, mining industry, rayon industry, metal rinse processes, tanning industry, fluidized bed bioreactors, textile industry, metal smelting, petrochemicals, paper manufacturing, and electrolysis applications. The heavy metal contaminated wastewater finds its way into the environment, threatening human health and the ecosystem. The heavy metals are non-biodegradable and could be carcinogenic [1-3].

The presence of these metals in water by improper amounts could result in critical health issues to living organisms. The most popular heavy metals are lead (Pb), zinc (Zn), mercury (Hg), nickel (Ni), cadmium (Cd), copper (Cu), chromium (Cr), and arsenic (As). Although these heavy metals can be detected in traces; however, they are still hazardous. Recent studies have focused on a particular method for heavy metal ions removal, such as electrocoagulation (EC), adsorption using synthetic and natural adsorbents, magnetic field implementation, advanced oxidation processes, membranes etc. These studies stood on the advantages and disadvantages of a specific method for wastewater treatment, including heavy metal removal. A complete picture of the heavy metals removal methods from wastewater resources has not been drawn yet [4, 5].

The present review comprehensively and critically discusses the available technologies to expel heavy metal ions from wastewater efficiently. Moreover, it is essential to choose the most applicable method based on the removal efficiency, chemicals added/adsorbents, initial concentration, optimal treated pH value, and other operating conditions. The removal of toxic metal ions from waste water is an important and widely studied research area in water treatment. It is essential to reduce the heavy metal concentration in effluents before they are discharged into the water bodies. Therefore, in research priority is given to regulating these pollutants at the discharge level. In recent years, the issues regarding disposal and treatment of effluent containing heavy metal pollutants has become a rising concern to the public. Several attempts have been made for their elimination, the objective being to design an effective and economic process. [6, 7]

Metal ions removal by ion exchange method is now considered one of the most promising techniques due to its cost effectiveness, eco-friendliness and rapidness. The recent developments in the synthesis of adsorbents containing polysaccharides in particular modified biopolymers derived from chitin, chitosan, starch, cellulose, guaran and cyclodextrin which are not only eco-friendly and cost-effective but are effective also in remediation of common effluents present in the wastewater [8].

Guar gum is a naturally occurring gum, also called Guaran, is a galactomannan. It is obtained from an annual pod bearing plant Cyamopsistetragonolobus or C. psoraloides, belonging to family Leguminosae. It is a high molecular weight carbohydrate. It is white to yellowish white in colour, odourless and is available in different viscosities and different granulometries. The guar gum molecule is a linear carbohydrate polymer with a molecular weight of the order of 2, 20,000. It is having a straight chain of D-mannose unit linked together by β (1- 4) glycoside linkage and D-galactose units are joined to it at each alternate position by an (1-6) glycosidic linkage. Hence, the Guar gum forms a rod like polymeric structure with a mannose backbone linked to galactose side chains, which are randomly placed on mannose backbone with an average ratio of 1:2 galactose to mannose. The polymeric structure contains numerous hydroxyl groups, which are treated for manufacturing different derivatives used for various applications in industries. The properties of galactomannan mainly depend upon their chemical features like chain length, Abundance of cis-OH group, steric hindrance, degree of polymerization and additional substitutions. Guarpolymerise resin used in removal of metal ions from dye industry waste water. The processing of guar gum is shown in figure-1.

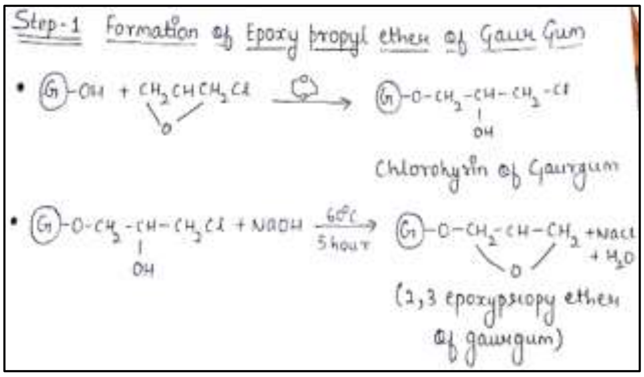
Figure-1: Processing of Gaur Gum

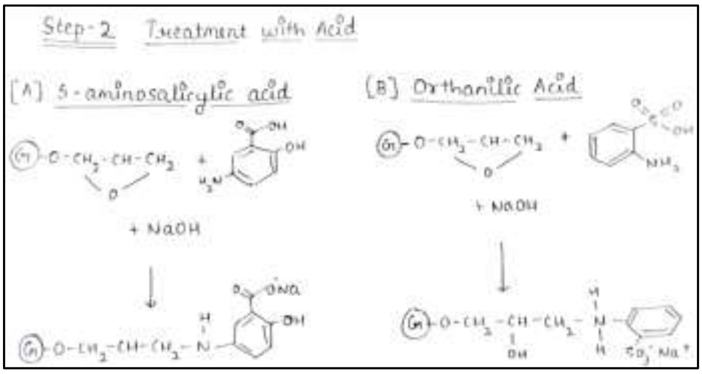
2. RESEARCH METHODOLOGY

2.1 Synthesis of GASA and GOAA resin

16 g Guar gam (0.2 mole anhydroglucose unit, (AGU) was taken in round bottom flask and slurred in 60 mL dioxane. While stirring the reaction mixture on a magnetic stirrer, 2.5 mL of 20% aqueous NaOH solution were added, followed by 5 ml of epoxychloropropane (chlorohydrin) and the mixture was stirred for 5 hrs. at 60°C. After keeping it overnight, the compound was filtered and washed with dioxane and ether. This form of functionalized guar gum can be stored at 25°C for long time. It can be activated by reaction with sodium hydroxide (NaOH) by converting the chlorohydrin into an epoxide group. In the dioxane suspension of guar chlorohydrin, dropwise 20% aqueous sodium hydroxide was added with stirring at 60oC. It was followed by addition of 11 g (0.1 mole) of 5-amino salicylic acid and orthanilic acid and stirring for 4 hrs. and left over night. Guar gum incorporating 5-amino salicylic acid and orthanilic acid group was filtered, washed with HCl-methanol and finally with ether and dried. The dark purple GASA and pale yellow GOAA resin were obtained.

2.2 Reaction mechanism





3. RESULT AND DISCUSSION

3.3.1 Moisture content and Elemental analysis:

1gm of resin, free from surface moisture was kept on a watch glass over ammonium chloride in vacuum desiccation for several days until a constant weight was obtained. The physicochemical properties like moisture content, nitrogen and sulphur content were studied according to the literature methods and the results are shown in table-1.

Table-1: Tabulated summary of observation for GASA and GOAA resins

S.No	Methodology	Formula employed	GASA	GOAA
1	Kjeldahl method	Nitrogen $=\frac{(b-a)\times N\times 14\times 100}{w}$		
		b = volume of NaOH for blank	14 mL	28mL
		a =volume of NaOH for resin	2mL	3.4mL
		w =weight of resin	0.8 g	0.9g
	Calculation	Nitrogen percent =	**42%**	**76.5%**
		N= normality of NaOH		
2	Messenger method	Sulphur$=\frac{w\times 32\times 100}{233\times W}$		
		w = weight of BaSO4.		0.38 g
		W =weight of resin		1 g
	Calculation	Sulphur percent =	—	**5.3%**
3	Moisture content	M.C.$=\frac{(W-w)}{W}\times 100$		
		W =weight of air dried resin	1 g	1 g
		w = weight of oven dried resin	0.8g	0.94g
	Calculation	Moisture content =	**20%**	**6%**

3.3.2 FTIR Studies

The FTIR (Fourier transform infrared) studies were done by a BRUKER Vertex 70 V in the range of wavenumber (400-4000cm^{-1}). Figure-2 shows the FTIR spectra 1 of GASA-Gaur gum 5-amino salicylic acid resin. The dominated peaks were found at 3386.9 cm^{-1} stretching band for OH, 2869 cm^{-1} for -CH stretching band of aliphatic (2800-3000 cm^{-1}),1449.8 cm^{-1} for C=C structure aromatic (1400- 1600 cm^{-1}),1343.11 cm^{-1} for -CH bend in aliphatic,1742.40 for C=O str. (1720- 1740 cm^{-1}) and 1503.33 cm^{-1} for –NH bend.

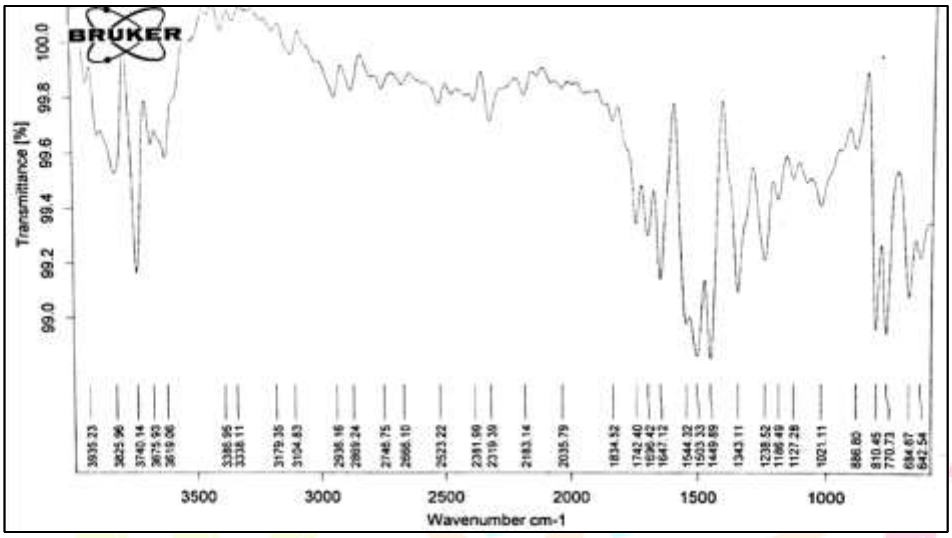


Figure 2: Spectra 1 of GASA-Gaur gum 5-amino salicyclic acid Resin

Figure-3 shows the FTIR spectra 2 of GOAA – Gaur gum Orthanilic Acid Resin. The dominated peaks were found at 3392.17 cm^{-1} stretching band for OH, 2869.78 for –CH stretching band of aliphatic (2800-3000 cm^{-1}), 1449.32 cm^{-1} (1400- 1600 cm^{-1}) for C=C structure aromatic, 2953.30 cm^{-1} for =CH in aliphatic,1509.30 cm^{-1} for –NH bend and1209.52 cm^{-1} for S=O

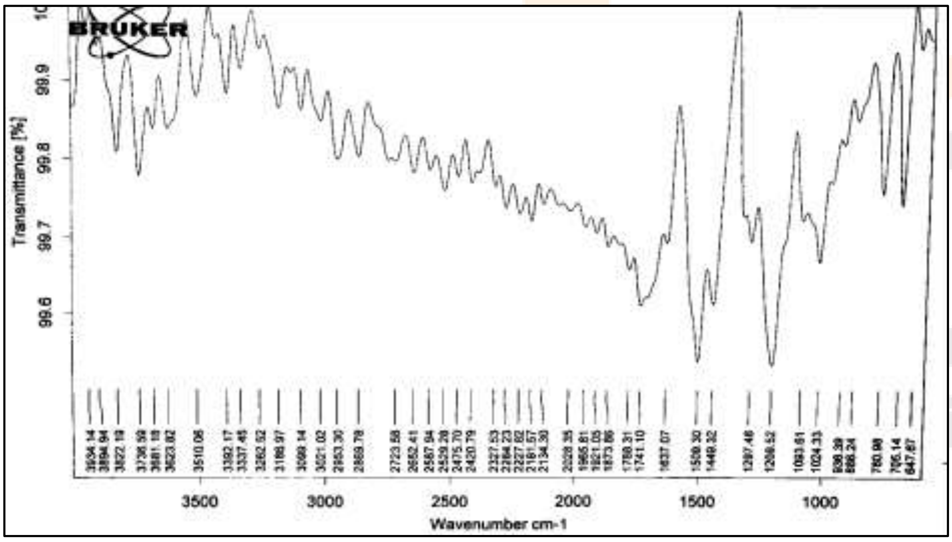


Figure 3: Spectra 2 of GOAA – Gaur gum Orthanilic Acid Resin

4. APPLICATION

4.1 Removal of toxic metal ions from effluent of mineral and metallurgical industries by batch method.

- **pH Adjustment**

15 ml of aliquots of effluent from Tripti dye and paint industry was taken and pH was adjusted to 4, 7, 9 using HCl and NH_3, and the pH was maintained using buffers.

- **Resin treatment**

1g of resin was added to each pH adjusted aliquot, and stirred constantly for 1 hour. After an hour filter through whatman filter paper no. 40 and make up the volume of filtrate to 50 mL.

- **AAS**

The filtrates were then analysed for toxic metal ions reduction in concentration from that of the untreated effluent **.**The effect of pH on its ion exchange capacity was thus determined, and also a comparison of the effectiveness of the two resins were established.

Table 2: GASA and GOAA RESINS

Metal ions	**Untreated Effluent**	**Resin Treated effluents at pH**					
	(ppm)	4 (ppm)		7 (ppm)		9 (ppm)	
		GASA	**GOAA**	**GASA**	**GOAA**	**GASA**	**GOAA**
Cadmium	0.14	0.08	0.091	0.00	0.002	0.05	0.03
Lead	0.42	0.23	0.187	0.06	0.003	0.09	0.021
Zinc	0.26	0.16	0.12	0.03	0.002	0.2	0.04

- **Graphical Interpretation**

Graphical representation of GASA and GOAA resins at different pH=4, pH=7 and pH=9 are shown in figure-4**.** At pH 4, both GOAA and GASA behave alike, GOAA being more effective. At pH 7, the extent of reduction of toxic metal is better than that in pH 4 by both the resins. GOAA reduces way more effectively than GASA. At pH 9, GOAA is more efficient than GASA**.**

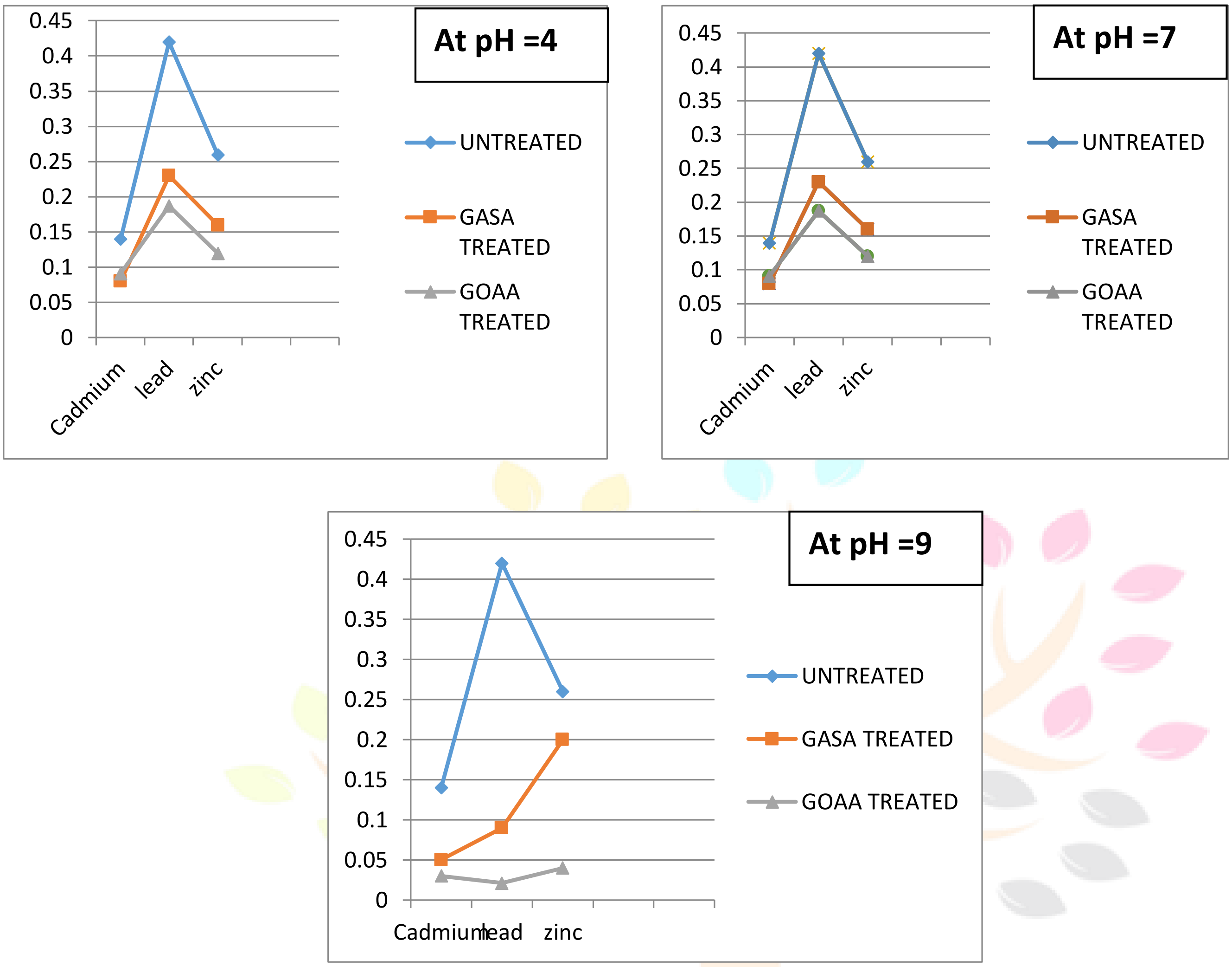


Figure 4- Graphical representation of GASA and GOAA resins at different pH=4, pH=7 and pH=9

5. CONCLUTION

The Guar Gum derived resins were found to reduce the heavy metal ions concentration at pH 7 for metals cadmium, lead, and iron to the level much below discharge limits .For the exchange of cation also, Guar Gum resin derived from orthanilic acid is way more effective than 5-amino salicylic acid. The Guar Gum powder was used since it is cost effective and readily available. It is also concluded that at pH 4, both GOAA and GASA behave alike, GOAA being more effective. At pH 7, the extent of reduction of toxic metal is better than that in pH 4 by both the resins. GOAA reduces way more effectively than GASA. At pH 9, GOAA is more efficient than GASA resin.

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