

Synthesis, Characterization and Analytical Study of Copolymeric Resin from Monomers of 2-thiobarbituric acid, Phenol and formalin

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ABSTRACT

Prepared by condensation polymerization of monomers, 2-thiobarbituric acid, phenol and formalin in alkaline medium.

The new copolymer was characterized by IR spectroscopy and thermal analysis. Also the copolymer was used as a cationic exchange resin for the study of several cations such as (Zn, Cd, Ni, Hg, Mn). The loading capacity of the resin towards the studied ions showing the following orders.



INTRODUCTION

The chelating polymers are one of the important classes of polymeric materials for their wide range applications, e.g. water treatment¹, conductors and semiconductors^{2,3}, extraction and separation of some expensive materials⁴, and the field of catalysis⁵, polymeric stabilizer⁶ and for some analytical applications⁷. The present work new a chelating polymer was synthesized and studied.

The reagent 2-thiobarbituric acid was used as a monomer for copolymerization with phenol and formalin by condensation polymerization to prepare cationic exchange resin. The prepared resin was identified using infrared spectroscopy and thermal analysis (TG and DTG). Two factors affecting chelating efficiency were studied, *i.e.* pH and treating time.

A three necked reaction vessel fitted with condenser efficient mechanical stirrer and thermometer was charged (0.72 g) of the monomer, (10 ml) of phenol, (100 ml) of formalin and (6g) of NaOH. The mixture was heated to reflux for 1:30 hours. The reflux was stopped and after cooling the pH was neutralized with diluted HCl filtered, washed with deionized water and dried at 60°C. and the resin was kept in vacuum desiccator.

2-Infrared spectroscopy.

Figs.(1) and(2) show the infrared spectra for monomer and

copolymer respectively which were measured using infrared spectrometer Philips model sp3-300s as KBr disc with range (200-4000 cm^{-1}).

3- Thermal analysis.

Figs.(3) and (4) show the TG and DTG for the monomer 2-thiobarbituric acid and the copolymer respectively which were measured by thermal analyzer, keito instruments inc. using heat rate $20^{\circ}\text{C}/\text{min}$.

4-Chelating efficiency

The chelating efficiency of the copolymer was examined toward the tested divalent ions (Mn^{+2} , Ni^{+2} , Cd^{+2} , Cu^{+2} , Hg^{+2} , Zn^{+2} , Fe^{+2}) from their diluted Solution using batch method by treating (0.1 g) of the copolymer with (25 ml) of (50 ppm) metal ion solution using mechanical shaker. The concentration of the metal ions was determined by using flame atomic absorption, shematzu model 630-12 using standard procedure. The effects of two factors namely pH and treating time on the resin capacity were studied.

5-Bonded metal recovery .

The bonded metal ions were recovered by treating the loaded resin with known concentration of the eluent (0.1, 0.5 and 1 N) HCl and monitoring the amount of metal ions in the eluent.

RESULTS AND DISCUSSION

. In order to confirm the Copolymerization infrared and Thermal analysis were used. The infrared spectrum of the monomer(Fig.(1)) show the bands $\nu_{\text{C-N}}$ (1100-1350 cm^{-1}) and $\nu_{\text{C-H}}$ (450-550 cm^{-1}) which may be due to wagging and bending of(- CH_2 -) *ON* using the reagent 2-thiobarbituric acid for qualitative analysis for several metal ions it shows no reaction for the metal ions of group I,II and.III therefore copolymerization of this reagent with phenol and formalin to introduced the S-H and O-H groups .respectively, these bands disappeared on copolymerization (Fig. (2)) due to restriction of their motion. On the TG ,DTG (Fig. (3) ,{ 4)) show also a complete difference between the curve of monomer and copolymer. exotherm peak of monomer (247.1°C) disappeared and the endotherm peak

appeared in copolymer.

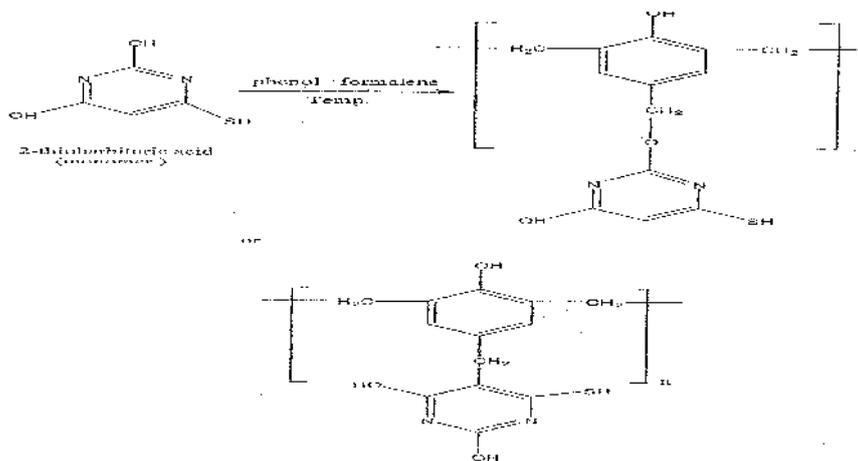
In addition the endotherm in monomer DTG curve 385°C was disappeared in the copolymer DTG curve.

The loading capacity of the copolymer were examined toward several metal _ ions at different pH which showed that the resin have an exchange ability , toward the ions with following orders (Ni~Cd>Mn>Hg>Zn) wheras the resin showed no response for exchange the metal ions(Fe,Cu) using different pH.

Fig .(5) show the maximum exchange ability of copolymer for Zn ion at pH=6 using treatment time of 2h, Fig.(6) show the maximum exchange ability of copolymer for Ni ion at pH=5 using treatment time of 2h, Fig(7) show the maximum exchange ability of copolymer for Cd ion at pH=8 using treatment time of 2h, Fig.(8) show the maximum exchange ability of copolymer for

Mn ion at pH=4 using treatment time of 6h, Fig.(9) show the maximum exchange ability of copolymer for Hg ion at pH=6 using treatment time of 6h The suggested structures for the new copolymer

are :-



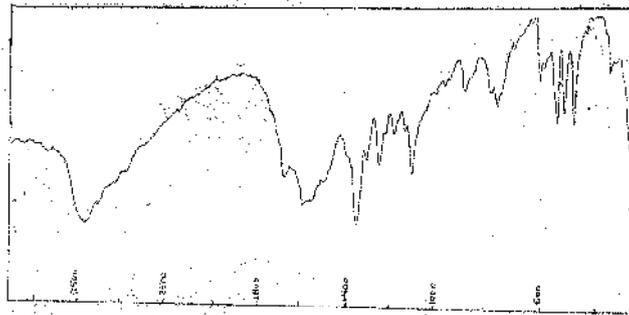


Fig.(1) Infrared spectrum of the monomer 2-bis(oxalobutanoic acid).

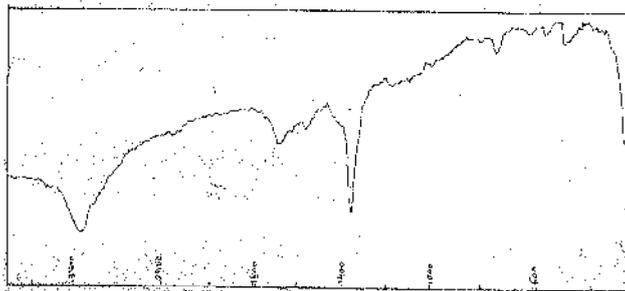
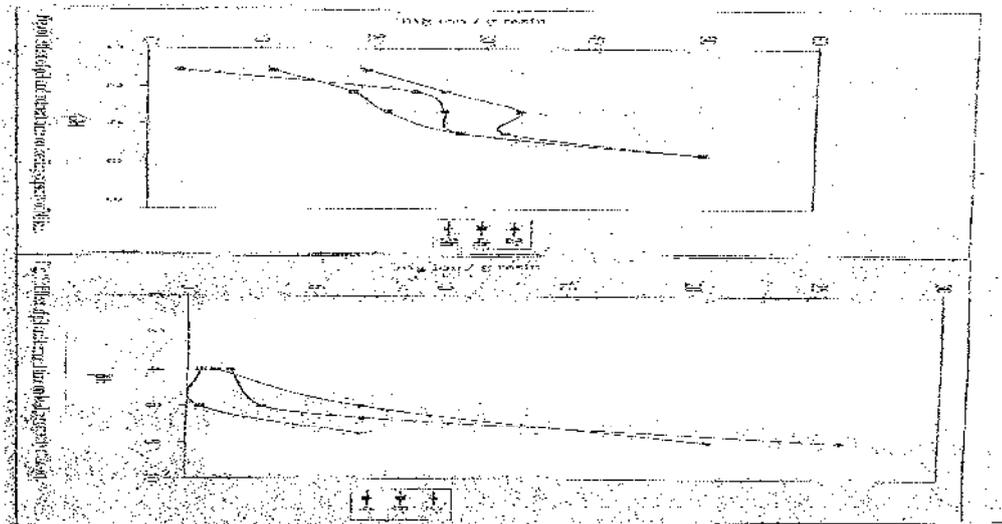
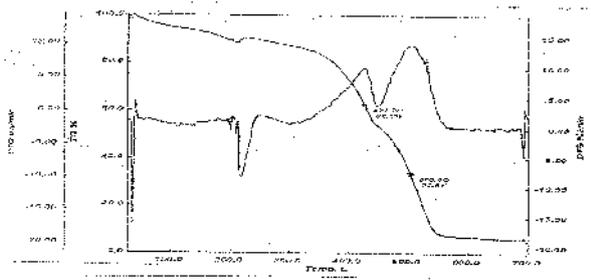


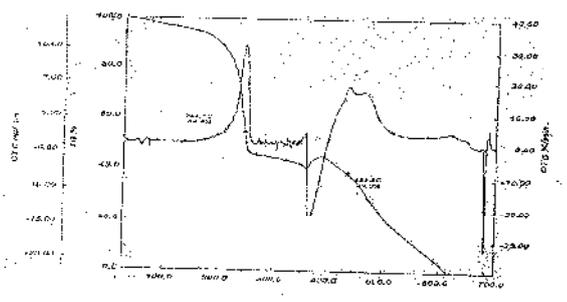
Fig.(2) Infrared spectrum of the prepared copolymer.

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Figure(3): TG and DTG of the monomer 2-thiobarbituric acid.



Figure(4): TG and DTG of the prepared copolymer.

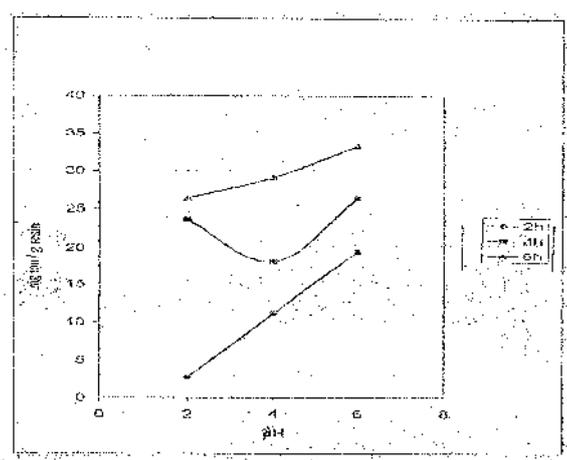
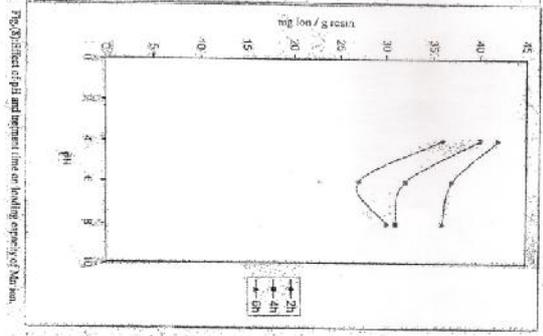
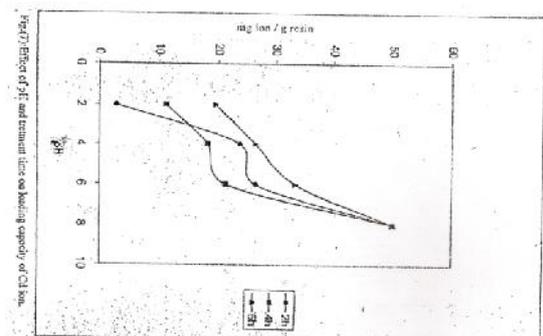


Fig (7): Effect of pH and treatment time on loading capacity of BA resin.

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تحضير وتشخيص ودراسة تحليلية لراتنجات محضرة من المونيمر

2 – *thiobarbituric acid , phenol and formali*

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الملخص:-

تم تحضير راتنج كوبولمري باستخدام البلمرة التكنيفية للمونيمرات 2 – ثايو حامض الباربيتوريك والفينول والفورمالين في محيط قاعدي حيث تم تشخيص الكوبوليمر المحضر باستخدام تقنية الاشعة تحت الحمراء والتحليل الحراري كذلك تم دراسة هذا الراتنج كمبادل أيوني للأيونات الموجبة (Mn , Hg , Ni , Cd , Zn) حيث اظهر الراتنج سعة تبادلية جيدة وكانت التبادلية للراتنج حسب كفاءته للترتيب الاتي :