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New method of the synthesis of aminophenolic ionites and study of ion sorption of non-ferrous metals and iodine

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Abstract

A simple method of obtaining of aminophenolic resins by condensation of dioxybenzene with hexamethylenediamine in the presence of formaldehyde was developed. The structure and properties of the reaction products, principal physical–chemical and sorption characteristics have been studied.

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1. Introduction

Phenolaminoformaldehyde resin Yarrezin B, usually used for the extraction of ions of heavy non-ferrous metals (zinc, lead, copper, cadmium, etc.) is produced in the Russian Federation. The resin based on 7-alkyl-8-hydroxyquinoline is well known as Kelex 100 or Lix 26 and is used to remove copper and gallium and to separate ions of toxic heavy metals from solutions of Ni(II), Cu(II), Zn(II), Cd(II), Hg(II) [1].

The Kazakh Republic is interested in similar sorbents by local production. Practically all hydrocarbons necessary for synthesis of such-like ion-exchange materials could be extracted from Kazakh oil.

2. Experimental

Organic reagents (resorcinol, pyrocatechol, hydroquinone) of the qualification "chemically

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pure" were preliminary dried in an desiccator over concentrated sulfuric acid to a constant weight. Hexamethylenediamine was stored in a desiccator over calcium chloride, calcinated at 102 °C.

The concentration of formaldehyde was determined following standard methods [2]. Formaldehyde of the concentration of 39.5–40.0% with the density of 0.9001–0.9003 g/cm^3 was used.

Catalysts – water solutions of ammonium hydroxide (28.0%, density of 0.898 g/cm^3), nitric (65.04%, density of 1.392 g/cm^3) and phosphoric acids (87.0%, density of 1.707 g/cm^3), hydrogen peroxide (30.0%, density of 1.086 g/cm^3) were prepared from the reagents of the qualification "pure for analysis" and "chemically pure".

Metal-containing solutions were prepared from the reagents (oxides, metals and salts) of the qualification "chemically pure" and "pure for analysis". The pH values of the solutions were maintained with the help of the solutions of mineral acids, ammonium and sodium hydroxides of the above-indicated qualification. pH value of the solutions and the potentiometric titration curves were registered on an automated Mettler-Toledo titrimeter.

Concentration of ions of non-ferrous and rare metals in water solutions was determined by an atom-absorption analysis on AAS 1N apparatus and by a photocolorimetric method on a KFK-2MP photoelectrocolorimeter [3]. The used reagents were of the qualification "chemically pure" and "pure for analysis".

Iodine-containing solutions were prepared by dissolution of elemental iodine in distilled water in the presence of sulfuric acid and sodium iodide. The necessary pH value of the solution was attained with the help of ammonium hydroxide or nitric acid. The initial concentration of elemental iodine in the solutions was equal to 200.4 mg/dm^3; the volume of the solution taken for sorption constituted 0.5 dm^3; the duration process is 24 h; dry ionite dose by weight ~0.1 g.

An estimation of a quantitative composition of the obtained aminophenolic resins was carried out on the basis of the data of elemental analysis.

IR-spectra of the initial, semi-, and final products were registered on a spectrophotometer SPECORD M80/M85 in tablets with KBr (200 mg KBr + 1 mg of the substance) and in the form of a thin film between the plates.

Thermograms were registered on a Paulik–Paulik Erdey derivatograph in the temperature range of 20–400 °C following the standard methods with the rate of heating of 5 °C/min and the rate of the movement of the diagram tape of 1 mm/min.

Synthesis of aminophenolic resins was carried out by a standard scheme of phenolformaldehyde resins obtaining [4]. The difference is being that dioxybenzenes instead of phenol and hexamethylenediamine as donor of active functional groups were used. Initiating additions of the acidic and basic type as well as hydrogen peroxide were used. The content of reagents in the reaction media varied (in mol) in the following ratios: DOB:FD:HMDA = 1.0:(3.5–11.5):(1.0–2.4), where DOB, dioxybenzene; FD, formaldehyde; HMDA, hexamethylenediamine. Synthesis and cross-linking were carried out in the temperature range of 25–180 °C. The reaction was performed for 1–3 h, thermal cross-linking – 5–24 h. After that polymers were ground to a homogeneous state, washed with distilled water up to a colorless reaction of filtrate, later rinsed by acetone, dried in a vacuum desiccator to a constant weight and stored in an desiccator over concentrated sulfuric acid.

The main sorption characteristics of the obtained resins were calculated as follows:

$$K_{\text{equil}} = \frac{(C_{\text{init}} - C_{\text{equil}}) \cdot V}{C_{\text{equil}} \cdot m}, \tag{1}$$

$$\varepsilon = \frac{(C_{\text{init}} - C_{\text{equil}})}{C_{\text{init}}} \times 100, \tag{2}$$

$$\text{SEC} = \frac{(C_{\text{init}} - C_{\text{equil}}) \cdot V}{m}, \tag{3}$$

where K_{equil} is a distribution coefficient, cm^3/g; ε is an ion extraction degree, %; SEC is the statistic exchange capacity, mg/g; m is the dry sorbent mass, g; V is the volume of the studied solution, cm^3; C_{init} is the element concentration in the initial solution, mg/dm^3; C_{equil} is the equilibrium (remaining) concentration of the extracted element in the solution, mg/dm^3.

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