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 Tamilla Muxtar kızı Naibova

 Azerbaijan State Oil and Industry University

 Candidate of Chemistry Sciences, Docent

Konul Qinyaz kızı Abbasova

Azerbaijan State Oil and Industry University Postgraduate student

SYNTHESIS SULFOCATIONITES BASED ON MODIFIED PHENOL-FORMALDEHYDE OLIGOMERS

Abstract: It has been realized modification of phenol-formaldehyde oligomers by benzamide and oksamide. It has been studied ways of obtaining sulfocationites on the basis of phenol-formaldehyde oligomers modified by benzamide and oksamide. To the comparative effect the changing of unmodified phenol-formaldehyde oligomer has been researched. At the end of modification reaction into each benzyl ring there was fed sulfonic acid qroup. Main indices of modified oligomers and sulfocationites have been researched.

It has been revealed that as a result of including additional funchional groups into modified oligomers basis characteristics of sulfocationites have been improved.

Key words: Phenol-formaldehyde oligomers, modified, benzamide, oksamide, sulphonation, ionite. Language: English

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Introduction

Ion-abundant oligomers are of great practical significance for various branches of national economy. In this respect obtaining sulphonication on the basis of modified phenol-formaldehyde oligomers (PhFO) has been rather actual [1-2].

For the synthesis of sulfonication we have obtained modified PhFO through well-known way having used benzamide and oxamide as a modifying agent.

PhFO modification has been realized in alkaline medium in the laboratory reactor provided with backflow condenser, thermometer and mechanical stirrer 1,15 mol of phenol, 1,45 mol of formaldehyde (in 37% solution) and 0,15 mol of 25% NH₄OH are placed in the reactor.

The mixture of the components being continuously stirred is heated up to 50-55^oC within 30-40 minutes and this very temperature 0,25 mol of modifying agent is partially added into this mixture. Then the temperature of the reaction mixture is heated

up to 90-95^oC and continuous stirring is going on of 60 minutes more. Meanwhile the reaction mixture grows turbid and is divided the upper layer is watery and the lower one is oligomeric. Oligomer is separated from water, washed in water to neutral reaction and dried in vacuum drying cabinet up to permanent mass. The output of modified oligomers makes up 70-75%. The obtained modified oligomers are well solved in acetone, dimethylformamide, tetrahydrofuran and dioxane.

In order to obtain comparative data in identical terms unmodified PhFO has been synthesized and researched [3].

To study physical-mechanical characteristics the obtained oligomers (modified by benzamide and oxamide as well as unmodified) were solidified in gradual temperature being heated up to 140°C within 4 hours [4, 122-123]. The solidification degree of the composition in accordance with its unsolvable part, determined through its extraction in the Soxhlet apparatus has made up accordingly 98,6%, 98,8% and



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92% [5, 31-36; 6, 89-92]. It has been revealed that, physical-chemical and physical-mechanical characteristics of modified oligomers significantly vary from identical features of unmodified PhFO (table 1).

It has been studied the swelling kinetics of modified and unmodified PhFO. Swelling is one of the available laboratory control methods of oligomer degree of structuring [7]. The higher the degree of structuration, the less is the probability of lower molecular solvent to penetrate into less molecular space of the polymer and the less is the swelling degree. The degree of swelling is changing throughout time. To estimate the ability of oligomers to swell, maximum degree value of swelling should be used. The degree of swelling, corresponding to the area of the horizontal area of the swelling diagram, represents the maximum degree of swelling [8, 728-740].

The graphic chart of the swelling degree of modified and unmodified PhFO in spirt-benzyl solution has been constructed.

Table 1. Physical-chemical and	d physical-mechanical indices of unmodified and modified Pl	hFO
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		Indices	Indices			
N⁰	Index denomination	Unmodified	Modified PhFO			
		PhFO	Benzamide	Oksamide		
1	Azotes content, % (weight)	—	6,3	11,2		
2	Content of free phenol, % (weight)	9,7	3,0	2,83		
3	Amount of methylol groups, % (weight)	11,2	8,95	9,24		
4	Amount of hydroxyl groups, % (weight)	17,5	12,52	12,68		
5	Adhesion strength, MPa	1,97	2,75	2,86		
6	Solidity according to Brinel, MPa	220	240	246		
7	Heat-resistance according to Vik, °C	105	156	160		

Figure shows that the swelling degree of modified PhFO is lower than the swelling degree of

unmodified PhFO. It is explained with the functionality growth of modified PhFO.

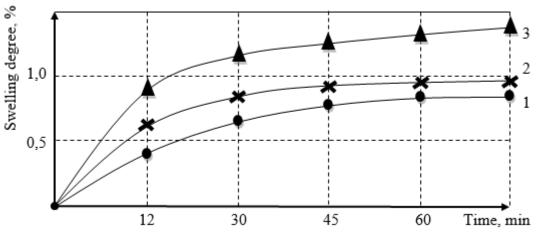


Fig. Dependance of the swelling degree in spirt-benzyl solution throughout time

- 1- PhFO unmodified
- 2- PhFO modified by benzamide
- 3- PhFO modified by oksamide

At the second stage it has been carried out sulfonation of functionalized oligomers. The sulfonation process was held in the laboratory reactor, of 250 ml capacity, provided with stirrer and backflow condenser. First an optimum amount of phenolformaldehyde oligomer modified by benzamide or oxamide and sulphuric acid of 98% are fed into the reactor. The reactor is heated untie oligomer is completely, the solution is cooled up to room temperature. Then immediately 37% water solution of formaldehyde is fed into the cooled reaction mass. Later this reaction mass is put into a special tank located in an oil bath at 11°C, where within 2 hours the solidification process goes on. After solidification the reaction mass is washed with water until it turns into clear water, then dried up and grained into 1-2 mm dimension particles.

Sulfocationite based on modified benzamide or oxamide phenol-formaldehyde oligomers products of black color, insoluble in water and hydrocarbons [9].

Main characteristics of the obtained sulfocationites are shown in table 2.



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Table 2. The main characteristics of sulfocationites on the basis of volume a	nd modified PhFO
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N₂	Sulphocationites	Functional qrup	Size of particles, min	Bulk weight, g/ml	Specific volume, ml/g	Static volume capacity, mg-ekv/g	Dinamic volume capacity, mg-ekv/g
1	Sulfocationite on basis of PhFO	- SO ₃ - OH	0,8-2,0	0,708	3,0	2,12	0,82
2	Sulfocationites on the basis of PhFO modified by benzamide	- SO ₃ - OH >NH >CO	1-2	0,62	8,4	2,96	0,98
3	Sulfocationites on the basis of PhFO modified by oksamide	- SO ₃ OH >NH >CO	1-2	0,52	8,4	3,62	0,96

It has been shown that as a result of presence of functional groups of various activity in the structure of sulfocathionites on the basis of functionalized phenolformaldehyde oligomers, their static capacity in comparison with sulfocathionites on the basis of unmodified phenol-formaldehyde oligomers increases.

Though the density of sulfocathionites obtained on the basis of modified PhFO rises, their swelling degree is higher, which is explained by partial dissolution of amine and amide groups in water during the volume process [10, 43-46]. Increased density and corresponding decrease of bulk volume of sulphocathionites on the basis of modified PhFO make easy regulation and their technical characteristics[11]. Obtained sulfocathionites have used for softening the water of definite solidity.

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