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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

SYNTHESIS AND İNVESTİGATİON OF PHENOL FORMALDEHYDE RESIN MODIFIED WITH LIGHT GAS OİL FRACTION OBTAINED FROM THE CATALYTIC CRACKING PROCESS

Speciality: **2304.01 – Macromolecular chemistry**

Field of science: chemistry

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GENERAL CHARACTERISTICS OF THE WORK

Relevance and degree of development of the topic. Due to the constant expansion of the application areas of high-molecular compounds and the increasing demands on their performance properties, one of the methods used to obtain polymer materials with useful complex performance properties is the modification of existing, industrially produced polymers, and the development of environmentally friendly synthesis and processing technologies for compounds distinguished by useful physical and mechanical properties in accordance with the principles of "green chemistry" is one of the perspective directions of the chemical industry of urgent importance in the modern era.

Phenol-formaldehyde (PF) resins, one of the first synthetic materials, are a polymer material that has been produced on an industrial scale since ancient times and has a wide range of applications.[1]

This is primarily due to the wide raw material base of PF resin, the fact that the production process is based on simple technology, and at the same time, the presence of reactive methylol and hydroxyl groups in the composition, and as a result, the possibility of modification with many different compounds. This provides the possibility of obtaining materials that are suitable for application in production areas such as coating formers, additives to rubber products and oils, plasticizing additives to concrete, surfactants, binders for layered materials, adhesives, abrasion-resistant abrasives, composite wood materials, and casting resins, which differ in useful indicators by purposefully changing the complex operational properties of the final product.

Currently, the demand for synthetic materials in various industries is constantly increasing and this issue is being solved by the synthesis of new high-molecular compounds with improved performance properties and the modification of high-tonnage

[1] Yang, L. Regeneration and utilization of waste phenolic formaldehyde resin: A performance investigation / J.Runt, M.C.Kuo[et al.] // Journal of Applied Polymer Science, – 2019. Vol. 136, №18, – p. 47445

polymers and oligomers with affordable, easily available functional components.[2]

In this regard, the incorporation of the gas oil fraction rich in mono- and bicyclic aromatic hydrocarbons obtained in the secondary refining, catalytic cracking process (CCP) of oil, into the synthesis process of phenol-formaldehyde resin is of great practical importance in obtaining a final product with improved operational properties and relatively low cost.

As is known, up to 30% of heavy oil residues are obtained during the oil refining process, and this includes the light gas oil fraction rich in aromatic hydrocarbons. The involvement of these compositions rich in aromatic hydrocarbons in the production of thermoplastic, thermoreactive polymer materials with complex useful operational properties, including the production of modified phenol-formaldehyde resins (MPFR), characterized by high quality indicators and economically efficient, is of prospective importance.

Modification of PF resins with light gas oil fraction obtained in the catalytic cracking process (CCP) allows the inclusion of aromatic hydrocarbons of various natures into the composition, while maintaining the reactivity of the macrochain. Depending on the oil production field and processing conditions, the composition and amount of aromatic hydrocarbons in the gas oil fraction varies, and as a result, the properties of the final product change.

Taking into account the above, this scientific research work was devoted to the promising direction of determining the synthesis conditions of MPFR with high yield by involving a mixture of aromatic hydrocarbons contained in the light gas oil fraction obtained from the CCP as a reaction component in the synthesis process of MPFR with the participation of new ionic liquid (IL) catalytic systems, studying its structure and physicochemical properties with modern analysis methods, obtaining various compositional compositions based on these resins, and determining their application areas.

Object and subject of research. The object of the research is

[2] Perederic, O.A. Life cycle analysis of phenol – formaldehyde resins substituted with lignin / O.A.Perederic, A.Mountraki, E.Papadopoulou[et al.] // Computer Aided Chemical Engineering, – 2020. Vol. 48, – p. 607-612.

the synthesis process of PFR with the light gas oil fraction of catalytic cracking, rich in aromatic hydrocarbons, obtained from the secondary processing of oil, catalytic cracking process at the Baku Oil refining Plant named after Heydar Aliyev. The subject of the research is the implementation of the modification process in the presence of ionic liquid catalysts of various compositions and the preparation of various composition compositions based on the obtained MPFR and the study of their physicochemical properties.

The purpose and objectives of the research. The research consists of developing effective synthesis conditions for PF resins with improved complex operational properties, modified with aromatic hydrocarbons contained in the light gas oil fraction obtained from the CCP, studying some physicochemical properties, including structure, thermal stability, electrical conductivity, obtaining various compositional compositions based on these resins, and determining a number of operational properties and application areas.

Research methods. The research carried out on the dissertation work is based on the application of modern analysis methods: IR and UV spectroscopy, Differential Thermal Analysis, electrophysical properties in the E6-13A therometer, microstructural morphology and chemical composition characteristics in the Hitachi S 3400 N Scanning Electron Microscope, and determination of the amount of aromatic hydrocarbons in the gas oil fraction by the sulfonation method according to GOST 15994-74, etc.

Basic provisions for defence:

– synthesis of reactive MPFR, modified with aromatic hydrocarbons contained in the light gas oil fraction obtained from the CCP of oil;

– implementation of the polycondensation process in the presence of new ionic liquid (IL) catalytic systems, development of optimal synthesis conditions for MPFR by investigating the effect of various factors on the process;

– comparative analysis of the modification process of MPFR in the presence of various IL-containing catalytic systems, study of the polycondensation process using a mathematical modeling program;

– study of the structure, thermal stability, electrical conductivity and other physicochemical properties of the synthesized MPFR using

modern analysis methods;
– study of the structuring process of MFFQ in the presence of cross-linking components such as urotropine and polyethylenepolyamine;
– preparation of compositions based on MPFR, epoxy resin and various organic-mineral compositions (bentonite, marble powder) and study of the structuring process;
– preparation of fuel briquettes and comparative study of their physical and mechanical properties using MPFR as a binding component and various wood wastes as fillers.

Scientific novelty of the research. The modification process of PF resin involves the determination of high-yield synthesis conditions for MPFR by involving a mixture of aromatic hydrocarbons contained in the light gas oil fraction obtained in the catalytic cracking process as a reaction component, obtaining various composite compositions, studying structuring processes using various methods, and determining the application areas of the final product:
– The modification process of PF resin with aromatic hydrocarbons contained in the light gas oil fraction of catalytic cracking was studied in the presence of IL catalytic systems of various compositions, and the synthesis conditions for the modified resin with a yield of 97% were determined, in which the IL catalyst containing N-methylpyrrolidonium hydrosulfate was more effective and efficient among these catalytic systems:
– For this purpose, the synthesis of IL catalytic systems based on N-methylpyrrolidone and orthophosphoric acid, containing hydro and dihydroorthophosphate in different molar ratios of components, was carried out, and for the first time, the modification conditions of PF resin with the mentioned mixture of aromatic hydrocarbons in the presence of these catalytic systems were determined;
– The implementation of the PF resin modification process with the participation of a regenerated IL catalytic system was studied, and it was shown that it is possible to reuse this type of catalytic systems;
– Structuring processes were studied in various ratios of MPFR and epoxy resin, in the presence of a cross-linking component, and it was determined that a fully structured product was obtained;
– Based on the study of the thermal-physical parameters of the

composition obtained on the basis of MPFR and bentonite as a filler, it was determined that these compositions are practically thermally stable up to a temperature of 200°C. The electrical conductivity parameters of these compositions in solid aggregate form and solutions of various concentrations (0.1-1.0%) in acetone were studied;

– The possibilities of obtaining fuel briquettes based on sawdust and shavings of pine and beech trees using MPFR as a binding component were studied. The conducted studies were approved by the patent of the Republic of Azerbaijan, "Method of obtaining fuel briquettes", invention I2024 0119.

Theoretical and practical significance of the research:

Based on the conducted studies, it was determined that the resin product obtained as a result of modification of PF resin with aromatic hydrocarbons contained in the light gas oil fraction is characterized by high thermal stability indicators, while maintaining reactivity.

The author's personal involvement. The personal participation of the author played a leading role in conducting experimental studies, analyzing and discussing the obtained results, developing and writing the strategy for implementing the dissertation work, publishing articles, and discussing the materials of the report and thesis at international conferences.

Approbation and application of research. The main results of the dissertation - III International scientific conference of young researches, Baku Engineering University, (Baku 2019); The İnternational Scientific conference "Actual Problems of Modern Chemistry" dedicated to the 90th anniversary of the Academician Y.H.Mammadaliev, Institute of Petrochemical Processes, (Baku, 2019); "International Scientific Conference on Innovative Development Prospects of Chemical Technology and Engineering", dedicated to the 70th anniversary of the city of Sumgayit (Sumgayit, 2019); Technology of organic substances: Materials of reports of the 84th scientific and technical conference dedicated to the 90th anniversary of BGTU and the Day of Belarusian Science (with international participation), (Minsk, 2020); International conference on actual problems of chemical engineering, dedicated to the 100th

anniversary of the Azerbaijan State of oil and Industry University, (Baku 2020); 6th International Turkic world conference on chemical science and technologies, (Baku, 2022); Respublican scientific conference dedicated to the 90th anniversary of academician Nadir Mir-Ibrahim oglu Seyidov, "Catalysts, Olefin-based oils", (Baku 2022); IV International Scientific and Technical Conference "Modern Achievements in the Field of Adhesives and Sealants: Materials, Raw Materials, Technologies" (Dzernisk 2023); Conference dedicated to the 90th anniversary of Academician Sahib Museib oglu Aliev, "Petrochemistry, Synthesis of Polyfunctional Monomers, Oligomers and Polymers", (Baku 2023); International Scientific Conference dedicated to the 60th Anniversary of the Department of Organic Matter and High Molecular Compound Technology of ASOİU, "Modern Problems of Macromolecular Compounds", Republic of Baku Technology (2024) and reflected in the materials of the international conference.

Publications. 20 scientific works have been published on the dissertation work. 9 of them are articles in well-known local and international journals, 1 patent, and 10 of them are reports and theses materials at international and local conferences.

Name of the organization where the dissertation work was carried out. The dissertation work was carried out in the laboratory of Texnology of organic Substances and High Molecular Weight Compounds department of ASOIU and in the laboratory of Polyfunctional monomers and oligomers, department of "Monomers, oligomers and catalysis" of the Institute of Petrochemical Processes named after academician Y.H. Mamamadaliyev.

The total volume of the dissertation, indicating the volume of the structural sections of the dissertation separately. The dissertation work is 148 pages (181410 characters) and consists of an introduction (12737 characters), 4 chapters (the first chapter - 55336 characters, the second chapter - 7135 characters, the third chapter - 57354 characters, the fourth chapter 46303 characters, the results - 2545 characters), a list of 182 references. The dissertation includes 40 figures and 24 tables.

MAIN CONTENT OF THE WORK

The introduction section, along with the justification of the relevance of the research carried out, provides an overview of the purpose of the work, the issues to be addressed, the scientific novelty of the work, its practical significance, and the approval of the conference materials reflecting the main results of the dissertation work. The introduction section consists of 12737 characters.

The first chapter provides an extensive and comprehensive literature review, including recent literature on the modification methods of phenol-formaldehyde resins, with references to monographs and books dedicated to this topic, journals with high impact factors, and other scientific works, consisting of 55,336 characters.

The second chapter consists of 7135 characters, giving the physicochemical characteristics of the reagents used in the synthesis of MPFR with aromatic hydrocarbons contained in the light gas oil fraction obtained in the catalytic cracking process of oil, as well as the synthesis methods and characteristics of intermediate products.

The third chapter describes in detail the synthesis processes of new ionic liquid catalytic systems, the synthesis of reactive MPFR based on the light gas oil fraction obtained in the CC process, the research conducted in the field of investigating the effect of various factors, the ratio of components, the composition and quantity of the IL catalytic system on this synthesis process, including the results of the mathematical modeling process of the synthesis process of MPFRs, and the results of the study of the thermal stabilities of phenol-formaldehyde resins synthesized under various conditions and composite compositions prepared on their basis, and consists of 57354 characters.

The fourth chapter presents the conditions for obtaining composite compositions using MPFR, mineral composition or organic composition wood dust as fillers, the results of obtaining fuel briquettes based on MPFRs, and the study of their physicochemical properties. In this chapter, the results of the study of the specific resistance and electrical conductivity of phenol-formaldehyde resin

synthesized under various conditions and composite compositions prepared on their basis are extensively commented and reflected in 46303 characters. At the end of the dissertation, the scientific results obtained from the research work carried out are given in 2545 references, a list of cited literature, and a list of abbreviated terms.

It is known that composite compositions prepared on the basis of PF resins were the first products of industrial importance, and even now they have not lost their practical importance in many areas of human activity. This is due to the wide raw material base of PF resins, the simplicity of the synthesis process, and the possibility of preparing various composite compositions, which ensures the relevance of this research work.

Taking into account the existing environmental problems, the modification of PF resins with aromatic hydrocarbons contained in the catalytic cracking product of petroleum, light gas oil fraction, was carried out in the presence of environmentally harmless, variously composed IL catalytic systems. This interest in the application of IM as a catalyst is determined by a number of unique properties inherent in them. In the conducted studies, variously composed IMs synthesized on the basis of sulfate and orthophosphoric acid, consisting of morpholinium hydrosulfate, N-methylpyrrolidonium hydrosulfate, diethylammonium hydrosulfate, triethylammonium hydrosulfate, N-methylpyrrolidonium hydroorthophosphate, N-methylpyrrolidonium dihydro-orthophosphate, were used as catalytic systems. The structures of the obtained IL compositions were determined by the IR spectral analysis method on the ALPHA brand Fourier spectrometer of the German company BRUKER.

In the studies, a light gas oil fraction with a boiling point of 190^{o}C and a boiling point of 345^{o}C was used as a modifier. It was investigated using a UV/Vis 6850 spectrometer, manufactured by JENWAY and by UV-spectral analysis determined that the content of aromatic hydrocarbons was 78.82% by mass.

The synthesis process of the modified phenol-formaldehyde resin was studied by adding phenol to the mixture of aromatic hydrocarbons in a 1.0-2:1 mass ratio, formaldehyde in a 1.0:0.8-1.0 molar ratio, and 1-5% of the catalyst, at a temperature of 96-98^{o}C and

a reaction time of 4 hours.

Table 1.

Effect of composition and amount of ionic liquid catalytic systems on the yield of phenol-formaldehyde resin modified with a mixture of aromatic hydrocarbons contained in light gas oil fraction

IL catalyst	Phenol:aromatic h/c mixture, ratio, mass	Phenol:formaldehyde ratio, molar	Catalyst amount, % mass	The reaction temperature, °C	The reaction time, hour	Yield of modified resin, % mass
Morpholinium hydrosulfate	1.0:1.0	1.0:0.8	2.0	96-98	4.0	69.5
	1.0:1.0	1.0:0.8	5.0	96-98	4.0	80.0
	1.0:1.0	1.0:1.0	2.0	96-98	2.5	86.7
	1.5:1.0	1.0:0.8	2.0	96-98	4.0	61.57
	2.0:1.0	1.0:0.8	2.0	96-98	4.0	64.15
	2.0:1.0	1.0:0.8	5.0	96-98	4.0	89.0
	2.0:1.0	1.0:1.0	2.0	96-98	3.0	86.75
	3.0:1.0	1.0:0.8	2.0	96-98	4.0	75.96
Diethy-lammonium hydrosulfate	1.0:1.0	1.0:0.8	5.0	96-98	4.0	36.0
	2.0:1.0	1.0:0.8	5.0	96-98	4.0	64.0
Triethylammonium hydrosulfate	2.0:1.0	1.0:0.8	2.0	96-98	4.0	49.3
	2.0:1.0	1.0:0.8	5.0	96-98	4.0	74.6
N-methylpyrrolidonium hydrosulfate	1.0:1.0	1.0:0.8	2.0	96-98	4.0	74.2
	1.0:1.0	1.0:0.8	5.0	96-98	4.0	86.0
	2.0:1.0	1.0:0.8	2.0	96-98	4.0	91.0
	2.0:1.0	1.0:0.8	3.0	96-98	4.0	92.6
	2.0:1.0	1.0:0.8	4.0	96-98	4.0	95.3
	2.0:1.0	1.0:0.8	5.0	96-98	4.0	97.0
	2.0:1.0	1.0:1.0	2.0	80,0	4.0	90.85
	2.0:1.0	1.0:1.0	2.0	96-98	4.0	94.7
	2.0:1.0	1.0:1.0	2.0	96-98	3.0	94.5
	2.0:1.0	1.0:1.0	2.0	96-98	2.5	94.0
N-methylpyrrolidonium hydroorthophosphate	2.0:1.0	1.0:0.8	3.0	96-98	4.0	71.4
N-methylpyrrolidonium dihydroorthophosphate	2.0:1.0	1.0:0.8	3.0	96-98	4.0	76.3

As can be seen from the table, when phenol is added to the mixture of aromatic hydrocarbons in the gas oil fraction in a 2:1 mass ratio, phenol to formaldehyde in a 1.0:0.8 molar ratio, and a reaction time of 4 hours, with an ionic liquid containing N-methylpyrrolidonium hydrosulfate as the catalytic system in an amount of 4% by mass relative to the total mass of components entering the reaction, the yield of the modified petroleum polymer resin is 95.3% by mass, but with an amount of 5% İL catalyst, the yield of the modified petroleum polymer resin is 97% by mass. Using 3% by weight of N-methylpyrrolidonium dihydroorthophosphate ionic liquid as a catalyst, the yield of modified phenol-formaldehyde resin under similar conditions is 76.3%.

As can be seen from the results obtained, when the polycondensation process is carried out in the presence of an IL catalyst, it is possible to obtain MPF resins of various compositions, and the process is more efficient than the process carried out in the presence of HCl acid. Thus, the effect of the ratio of various factors-components, reaction temperature and duration on the polycondensation process in the presence of IL catalytic systems was investigated, and the synthesis conditions of a new composition MPFR, characterized by a complex of useful physicochemical properties with high yield were determined.

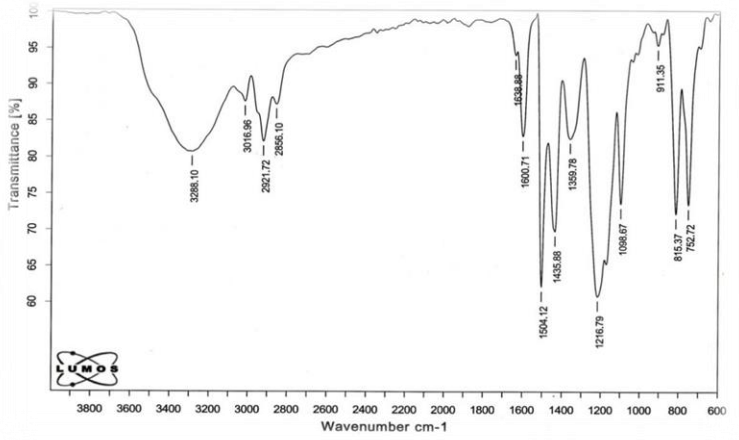
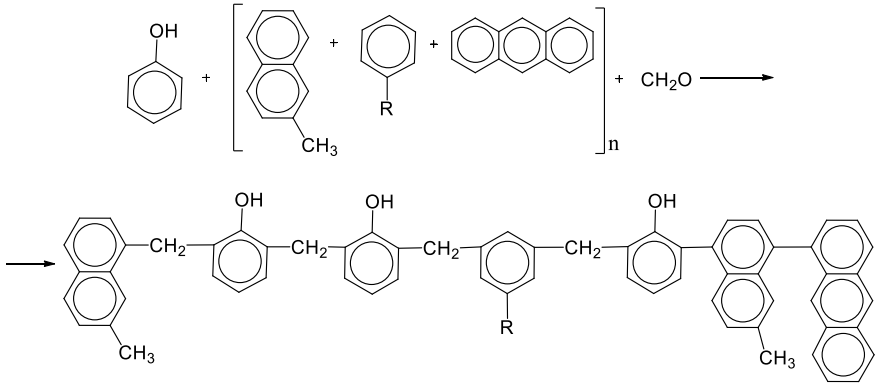


Figure 1. IR spectrum of MPFR synthesized in the presence of an ionic liquid catalytic system N-methylpyrrolidonium hydrosulfate.

In the IR spectrum of the synthesized MPFR sample, the absorption bands reflecting the deformation and valence vibrations of the C-H bond in the CH_2- group are observed at (1360, 1435 cm^{-1}); (2856, 2921 cm^{-1}), the absorption bands reflecting the deformation vibrations of the alkyl substituents of the benzene ring (752,815,911 cm^{-1}), and the absorption bands reflecting the deformation and valence vibrations specific to the C-H bond in the benzene ring are observed at wavelengths of 1504, 1600 cm^{-1}, 3016 cm^{-1}. The absorption bands observed at wavelengths of 1042 cm^{-1} and 1096 cm^{-1}, including 1216 cm^{-1}, characterize the presence of the C-O bond in the methylol and phenol fragments, respectively. In addition, the valence vibrations observed in the spectrum at a wavelength of 3288 cm^{-1} confirm the presence of an H-O bond in the hydroxyl group of phenol, which in turn confirms the presence of phenol fragments in the obtained resin.

The synthesis reaction and structure of MPFR can be imagined as follows:



The environmental problems that have arisen in modern times emphasize the need to avoid waste products in production and processing processes and the reuse of reagents. Taking this into account, the regeneration and reuse possibilities of the N-methylpyrrolidonium hydrosulfate IL catalytic system used in the studies were investigated and it was determined that the yield of the intended or desired product was 76% by mass.

Mathematical modeling of the synthesis process of MPFR was based on the results obtained from the implementation of the polycondensation process in the presence of ionic liquid catalysts of

various compositions HCl and MFHS, NMPHS. The effect of various factors on the yield of the target product was investigated and by repeating the experiment at the determined optimal values of the input parameters, it was determined that the yield of the target product was 98.5% in the presence of the ionic liquid catalyst NMPHS.

Thus, the developed mathematical model, developed in the form of a regressive polynomial, adequately reflecting the experimental data, allowed us to determine the optimal values of the input parameter that ensure a high yield of the target product.

Considering that high-molecular compounds containing a sufficient number of heterocyclic and aromatic fragments in the macrochain are thermally stable and resistant to oxidation, the thermal stability of MPFR was studied in the thermal analysis device "Jupiter STA 449 F3" of the German company "NETZSCH" in the temperature range of 25-700ºC and the heating rate was 10ºC per minute.

Table 2

Mass loss of unmodified and modified PF resin samples

MPFR samples	Mass loss, %, at temperature, °C										Residual mass/at temperature, °C
	250	300	350	400	450	500	550	600	650	698	
N1	8.1	13.2	19.3	32.2	42.6	46.8	50.2	54.3	58.1		42.11% /647.9
N2	-	-	2.8	7.6	14.7	22.9	26.3	33.4	43.6	52.6	47.44% /698
N3	6.8	12.3	16.7	24.3	31.6	36.2	43.3	52.7	62.4	74.8	25.21%/ 697 .9
N4	-	7.1	11.7	16.3	22.4	28.1	35.8	44.1	48.7	50.49	49.51% /697.8
N5	2.3	3.8	5.2	7.4	17.2	29.6	38.1				59.09%/596.3
N6	-	-	-	1.7	12.4	24.1	30.4	36.4	43.4	50.5	39.09%/697.9

In order to investigate the dependence of the thermal stability of the synthesized MPFR on the composition, the thermal stability of the structuring products (N5, N6) of the MPFR samples synthesized under analogous conditions at a 1:1.1 molar ratio of phenol to formaldehyde, an equal mass of unmodified PF resin (N1) and a mixture of aromatic hydrocarbons of phenol and formaldehyde at a 1:0.8 molar ratio, 2% and 5% mass amounts of catalyst (N2 and N3), and 5% mass amount of IL (N4), as well as unmodified (N1) and modified resin samples

(N3) in the presence of 10% mass urotropine was studied (Table 2).

It was found that the unmodified resin sample (N1) is characterized by relatively low thermal stability compared to the modified resin sample (N2) under similar conditions, and the mass loss at 350°C is 2.8% mass versus 19.3% for the MPFR sample, and at 650 °C it is 58.1% versus 43.6% mass. As the amount of IL catalyst used in the polycondensation process increases, the thermal stability of the obtained modification (N3) decreases, and the mass loss at 698 °C is 74.8%.

Thus, the conducted studies have a positive effect on obtaining products characterized by relatively high thermal stability and adjusting the thermal stability indicators of the resin, reducing the cost of the final product, modification, and production.

The electrical conductivity of the MPFR sample was determined in solution and in the solid state was determined by turning it into a tablet. As the concentration of the resin sample in acetone increases from 0.1% to 0.7%, the electrical conductivity of the solution increases, and with a further increase in concentration, a decrease in electrical conductivity is observed, and the specific electrical conductivity of the resin in acetone at a concentration of 1.0 is $2.12 \cdot 10^{-5}$ $Om^{-1} \cdot m^{-1}$.

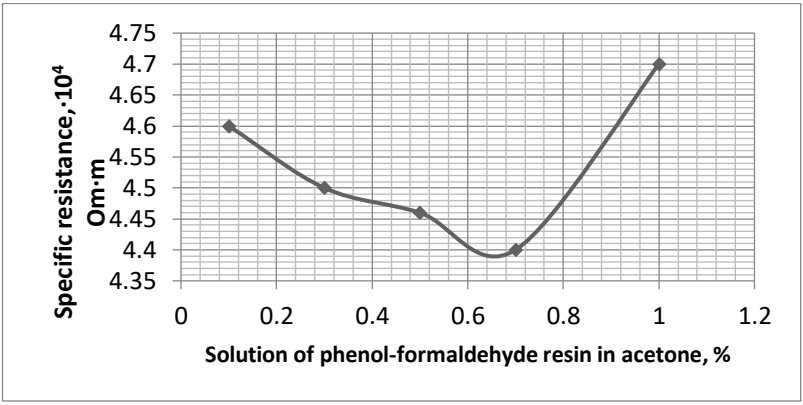


Figure 2. Graph of the dependence of specific resistance of samples of modified phenol-formaldehyde resin in acetone at various concentrations on concentration.

The results of the studies show that the electrical conductivity of MPFR increases with increasing temperature, starting from room temperature (23°C) and ending at 53°C, and then stabilizes.

Table 3

Electrical conductivity of a tablet prepared from a MPFR sample at different temperatures

Temperature, °C	Resistance,R, Om	Specific resistance, ρ, Om·m	Specific electrical conductivity,σ,$Om^{-1}·m^{-1}$
23	10^6	$6.9 \cdot 10^6$	$1.4 \cdot 10^{-8}$
28	$9.8 \cdot 10^5$	$6.76 \cdot 10^6$	$1.47 \cdot 10^{-7}$
33	$9.4 \cdot 10^5$	$6.48 \cdot 10^6$	$1.54 \cdot 10^{-7}$
38	$9.3 \cdot 10^5$	$6.42 \cdot 10^6$	$1.55 \cdot 10^{-7}$
43	$9.2 \cdot 10^5$	$6.35 \cdot 10^6$	$1.57 \cdot 10^{-7}$
48	$9.0 \cdot 10^5$	$6.21 \cdot 10^6$	$1.61 \cdot 10^{-7}$
53	$8.88 \cdot 10^5$	$6.13 \cdot 10^6$	$1.63 \cdot 10^{-7}$
58	$8.81 \cdot 10^5$	$6.08 \cdot 10^6$	$1.64 \cdot 10^{-7}$
63	$8.8 \cdot 10^5$	$6.07 \cdot 10^6$	$1.64 \cdot 10^{-7}$
68	$8.8 \cdot 10^5$	$6.07 \cdot 10^6$	$1.64 \cdot 10^{-7}$
73	$8.8 \cdot 10^5$	$6.07 \cdot 10^6$	$1.64 \cdot 10^{-7}$

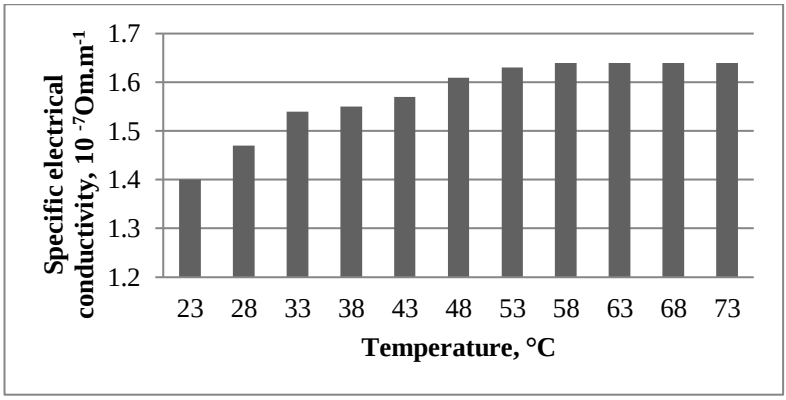


Figure 3. Temperature dependence diagram of the electrical conductivity of a tablet prepared from a MPFR sample

This result gives us the basis for changing the physicochemical properties of the modification obtained from the modification of

phenol-formaldehyde resin with a mixture of aromatic hydrocarbons contained in the gas oil fraction in one direction or another, according to the purpose.

Taking into account the above, research has been carried out to obtain and study various composite compositions based on MPFR with alkylaromatic hydrocarbons.

Based on the conducted studies, it was determined that the thermal structuring of the composite composition obtained in the equal mass ratio of MPFR to epoxy resin (1:1) is observed at a temperature of 160ºC, and the degree of structuring within 1 hour is 86.5% by mass. When the amount of epoxy resin in the composition is reduced to 10% by mass, the yield of the gel fraction at a temperature of 180ºC is 62.8% by mass within 3 hours, and 66% by mass within 5 hours.

At a 10:90 mass ratio of MPFR to epoxy resin, thermal structuring was not observed even at 180ºC.

The structuring process in the presence of the cross-linking component urotropin was carried out at temperatures of 100ºC, and in the presence of PEPA at 25-90ºC, and the decisive role of the ratio of components was once again confirmed. As the amount of epoxy resin in the composition increases from 10% by mass to 60% by mass, the yield of structured resin increases and at 100ºC with 10% by mass of cross-linking component, it was 99% by mass within 1 hour. When PEPA is used as a cross-linking component at room temperature, the amount of cross-link part mass is 93% by mass, and at 90°C it is 96.4% by mass in just 30 minutes.

It was investigated the possibility of obtaining coatings with decorative properties resistant to air, light, water and ultraviolet rays based on composite compositions obtained with the participation of synthesized MPFR, epoxy resin and 7% and 9% mass PEPA as a cross-linking component.

Various additives and pigments are used in various industries to obtain coatings with decorative and protective properties. In the studies conducted taking into account the above and the ability of the dry of these compositions in a short time at room temperature, the production of decorative coatings on marble slabs was studied by adding 4% by mass of Fe_2O_3, FeO and Cr_2O_3 pigments belonging to

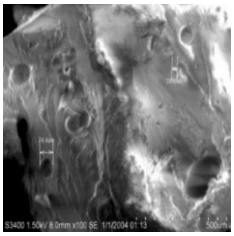
the achromatic group to 15-30% solutions of the MPFR sample in alcohol and acetone.

Figure 4. Coatings obtained on the surface of marble slabs based on MPFR: pigment- Fe_2O_3 - (a), FeO -(b), Cr_2O_3 - (c)

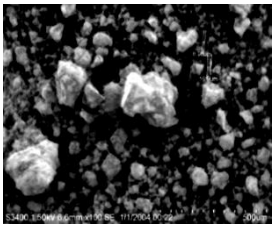
It has been shown that the color shades of the coatings obtained vary depending on the composition of the pigment used, and it is possible to obtain decorative coatings with different appearances and purposes based on MPFR.

It is known that adding fillers of various nature to composite compositions reduces the cost of the final product and improves its operational properties. The yield of the compositions obtained by adding 5% by mass of filler and 10% by mass of PEPA to a mixture of ED-20 and MPFR in equal mass ratios and structured for 4 hours at room temperature was 96.85% by mass when marble powder was used as the filler component, and 92.67% by mass when wood sawdust was used.

In addition, the preparation of a composite sample based on MPFR and bentonite as a filler was carried out by adding 3% mass of mineral filler during the synthesis process.The structural structures of the obtained composite samples were examined on a Hitachi-S 3400N Scanning Electron Microscope and the surface images obtained through SEM analysis of the mentioned samples were taken under the conditions of 1.50 kV × 7.3 mm 100 SE.



(1)



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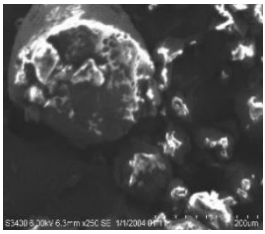

(3)

Figure 5. Microscopic image of MPFR (1), bentonite (2), and composite obtained by adding bentonite (3).

SEM analysis of the samples shows that the sizes of the components are larger, ranging from 27 to 139 μm, compared to the composite sample prepared based on MFFQ and bentonite, and the sizes of the FF resin and bentonite particles vary from 9 to 17 μm.

An interesting point is that in the composite sample prepared based on these two components, it is observed that irregularly distributed parts with sizes ranging from 22 to 52 um are formed. This allows us to assume that as a result of the distribution of bentonite particles between the chains of the obtained MPFR chains during the preparation of the composite sample with the addition of bentonite, numerous parts consisting of relatively small binder and filler are formed. As a result, the obtained composites containing bentonite are characterized by higher mechanical strength than MPFR.

IR-spectral analysis of composite compositions obtained by adding MPFR and bentonite or halloysite as mineral fillers was carried out and it was determined that they were characterized by absorption bands specific to the modified resin.

Table 4.

Optical densities of fragments specific to MPFR and composite compositions based on it

Samples	D_{1211}(OH) Phenol fragment	D_{1503}(C-H) Benzene ring	D_{1600}(C=C) Benzene ring
MPFR	0,455	0,388	0,151
MPFR+ 3% bentonite	0,206	0,220	0,098
MPFR+ 3% halloysite	0,250	0,227	0,089

The electrical conductivity of an organic-mineral composite prepared by adding MPFR and 3% bentonite by mass to the composition was studied and it was determined that the electrical insulation properties of the resin are improved by the application of mineral filler.

Table 5.

Electrical conductivity and resistance of MPFR and organic-mineral composite prepared on its basis

Sample	R, Om	ρ, Om·m	λ,$Om^{-1}\cdot m^{-1}$
MPFR	$2{,}8\cdot10^{7}$	$1{,}44\cdot10^{10}$	$6{,}9\cdot10^{-6}$
MPFR+ 3% bentonite	$2{,}2\cdot10^{10}$	$8{,}3\cdot10^{10}$	$1{,}2\cdot10^{-7}$

In the next stage of the research, organic-mineral composite compositions of various compositions were obtained by changing the ratio of components based on MPFR and epoxy resin and mineral filler bentonite, and their physical and mechanical properties were comparatively studied.

The components included in the composition of the mentioned composite samples were completely mixed and brought to a homogeneous state, and then hot pressed for 45 minutes under a pressure of 180-240^{o}C kg/cm^2 (table 6).

Table 6.

Composition of composite samples based on modified phenol-formaldehyde resin

Components	Composition of composite samples based on MPFR, %(mass.)		
	№1	№2	№3
MPFR	23	28	67
Epoxy resin	10	5,0	—
Bentonite	67	67	33

Table 7 shows the physical and mechanical properties of composite samples prepared with the indicated composition.

As can be seen, composite compositions prepared based on modified PF resin are characterized by high strength properties and water resistance compared to phenol-formaldehyde resin.

Table 7.

Physical and mechanical properties of composite compositions prepared on the basis of modified phenol-formaldehyde resin

Composite samples	degree of hardening, % (mass)	Impact resistance MPa	Compressive strength, MPa	Water absorption capacity 150 hours, %
MPFR №1	98,2	24,7	34,0	—
MPFR №2	98,6	20,9	37,2	0,25
MPFR №3	98,2	21,0	38,2	0,6
PFR	99,8	5,0-10,0	—	1,1

The depletion of traditionally used energy sources and the associated increase in their price, as well as the elimination of environmental problems caused by waste products that pollute the environment, have aroused great interest in the production and application of fuel briquettes molded in a certain shape as a promising alternative to traditionally used wood-based biofuels (wood and sawdust, peat, oil-based fuels, etc.). It should be noted that this type of fuel briquettes has a number of advantages, such as the simple method of obtaining it, its ecologically harmlessness despite the use of waste products, etc.

In the conducted studies, was investigated the production of fuel briquettes using MPFR as a binding ingredient.

For this purpose, briquettes of 10g, were prepared by pressing, using MPFR binder, beech, and pine sawdust as filler components, and after the various sized wood sawdust taken were completely mixed with MPFR, the mixture was pressed under a temperature rising from 50ºC to 100ºC and a pressure of 50-150Mpa.

It was determined that the briquettes obtained by adding 0.5; 0.7 and 1.0% of MFFQ as a binder to the beech sawdust taken as a filler were characterized by a density of 1.8-2.0 g/cm3, a compressive strength of 2.2-3.5 MPa, and a heat capacity of 9300 kcal/kg.

The burning time of these briquettes increased by 1.9-14.1-30.7% compared to briquettes prepared by pressing without any

additives (15.3 minutes) and was 16.0; 17.46; 20.0 minutes, respectively. The same situation is observed in briquettes obtained by adding 0.5% mass of beech charcoal (BC) and 0.5% mass of MPFR to pine and beech sawdust. The burning time of these briquettes was 26 minutes compared to 15.3 minutes for the unfilled beech briquettes. The burning time of the fuel briquette sample obtained using pine sawdust as a filler was 20.2 minutes (Table 8).

Table 8.

Combustion characteristics of briquettes based on beech and pine sawdust

№	Composition of briquettes	Time until ignition, min	Full ignition time, min	Combustion time, min.
	Beech shavings (without additives)	1.40	6.50	15.30
	Beech + 0.5% MPFR	1.15	5.2	16.0
	Beech + 0.7% MPFR	1.20	4.9	17.46
	Beech + 1% MPFR	1. 9	4.50	20.0
	Beech + 0.5% MPFR+ 0,5% BC	0.28	4.0	26.0
	Pine + 0.5% MPFR + 0,5% BC	0.5	3.6	20.2
	Pine + 0.7% MPFR	1.5	5.4	17.3
	Pine + 1% MPFR	22.2	5.1	18.4
	Pine + 0.5% MPFR	1.3	5.8	16.9

As a continuation of the research work, the effect of the size of the wood sawdust taken as a filler on the combustion performance of the prepared fuel briquettes was also investigated. For this purpose, the wood sawdust used as a filler was first passed through a sieve (TGL 7354) with a diameter of 20 cm and pore sizes of 0.4 mm, 1.6 mm and 2.0 mm. Briquettes were prepared under pressure by hot pressing under the specified conditions using the obtained sawdust of pine and beech trees of different sizes as a filler component and MPFR as a binder component.

In this case, the compressive strength index changes analogously to the density index of the resulting fuel briquette. It has been shown

that the combustion heat of the resulting fuel briquettes varies depending on the size of the sawdust and the amount of filler.

Table 9.

Combustion characteristics of briquettes obtained using beech and pine sawdust of different sizes as filler

№	Sizes of wood chips, mm	Amount of binder component, % mass	indicators of the briquette					
			Density, g/cm3	Compressive strength, MPa	Warm-up time, minutes	Burning time, min.	Heat of combustion, kcal/kg	Residual ash content, % mass (to
N 1	Beech tree 0.4	0.7	1.2	3.0	0.27	20.43	9500	1.07
N 2	Beech tree 1.0	1.0	2.0	3.5	0.28	26.0	10000	1.2
N 3	Pine tree 2.0	0.7	2.0	2.5	1.18	25.4	9700	2.0
N 4	Beech tree 2.0	0.7	1.8	3.0	1.2	25.0	10000	1.2
N 5	Pine tree 1.6	0.7	1.9	2.8	1.18	25.4	9800	1.8
N 6	Beech tree 0.8	0.5	1.4	2.2	1.4	16.3	9300	1.4
N 7	Beech tree 1.5	0.5	1.9	3.5	0.28	26.0	11000	1.05

Thus, by reducing the amount of filler from 0.7% by mass to 0.5% by mass, the fuel briquette obtained on the basis of beech sawdust with a size of 1.5 mm is characterized by a relatively high combustion heat index (11000 kcal/kg). The burning time of this sample is also high, 26.0 minutes, and the amount of ash obtained during combustion is 0.105% by mass, that is, a total of 1.05% g [20].

Fuel briquettes obtained using pine sawdust as a filler and the same amount of binder, regardless of the sawdust size, are characterized by practically very similar density (1.9-2.0 g/cm3), compressive strength (2.5-2.8 MPa), ignition time (1.18 minutes), burning time (25.4 minutes), residual ash content (1.8-2%) and combustion heat index (9700-9800 kcal/kg).

Thus, the initial results obtained indicate that the quality

indicators of fuel briquettes obtained on the basis of MPFR and various types of wood chips depend on the amount of binder, type of wood, and size of the sawdust.

It is known that the main requirement for fuel briquettes is that they do not change their shape under the influence of humidity during storage.

Taking this into account, the degree of moisture absorption from the air of briquettes obtained on the basis of wood waste and a binder composition in the amount of 0.7% by mass was investigated.

When the prepared briquettes were stored at room temperature for 3 months, it was determined that the mass of the briquettes prepared using beech sawdust increased by 0.99% by mass, and the mass of the briquettes prepared from pine increased by 0.97% by mass, and no changes were observed in the visual appearance of the briquettes.

Table 10.
The rate of moisture absorption from the air by briquettes

№	Composition of briquettes	Initial mass	Mass after 3 months	Mass increase, %
	Beech (1.6 mm) +0.7% MPFR	10.201	10.302	0.990
	Pine (1.6 mm) +0.7% MPFR	10.077	10.175	0.973

Thus, the initial results obtained indicate that the use of MPFR as a binding component in the preparation of composite materials for various purposes, including fuel briquettes based on wood waste, is possible and has prospective significance.

RESULT

1. The polycondensation process of a mixture of phenol, formaldehyde, and aromatic hydrocarbons contained in the light gas oil fraction obtained in the secondary refining and catalytic cracking process of petroleum in the presence of ionic liquid catalytic systems of various compositions was studied, and the synthesis conditions of a new composition MPFR were determined [1-6,15].

2. The effect of various factors (ratio of components, nature and amount of ionic liquid catalyst, reaction temperature, reaction time, etc.) on the polycondensation process was studied, and the synthesis conditions for MPFR with a mass yield of 97% were determined: mass ratio of phenol to aromatic hydrocarbons 2:1, mole ratio of formaldehyde 1:0.8, amount of ionic liquid catalyst - N-methylpyrrolidone hydrosulfate - 5% mass, reaction temperature 98°C, duration - 4 hours [2,12].

3. Composite compositions were prepared based on MPFR and epoxy resin in different ratios of components, and structuring was carried out in the presence of polyethylenepolyamine and urotropine. It was found that when the structuring process was carried out in the presence of urotropine as a cross-linking component, at a temperature of 100ºC, and in the presence of PEPA at a temperature of 90ºC for 0.5 hours, a fully structured spatially structured polymer was obtained [7,14].

4. Thermogravimetric analysis of composite samples based on MPFR and epoxy resin was performed and found that, MPFR thermally stable compared to composite compositions prepared based on unmodified phenol-formaldehyde resin [7,8].

5.The electrical conductivity of composite compositions obtained in the presence of MPFR and mineral filler bentonite was studied using the samples in solid aggregate form and solutions of various concentrations (0.1-1%) in acetone, and it was determined that it is possible to adjust the electrical insulation properties of the obtained compositions [18].

6.The conditions for obtaining organic mineral composites of various compositions based on MPFR and epoxy resin taken in equal

mass ratios were determined. It was shown that when a mineral filler (bentonite) is added to the reaction mixture during the resin synthesis process, a more homogeneous composite composition is obtained [13,19].

7.The softening temperature of the synthesized petroleum polymer resin samples obtained by adding bentonite as a mineral filler and bentonite was determined in the Boeutus apparatus. It was determined that the softening temperature increased relatively as the amount of phenol in the composition increased, and the composite composition obtained by adding bentonite did not melt even at a temperature of 200°C [17].

8.The possibility and conditions of obtaining fuel briquettes using MFFQ as a binding component and pine and beech sawdust as fillers were studied. It was shown that briquettes obtained by adding MPFR in amounts of 0.5; 0.7 and 1.0% are characterized by a density of 1.4-2.0 g/cm^3, a compressive strength of 2.2-3.5 MPa, a heat capacity of 9300-11000 kC/kg and a burning time of 16.3-26 minutes [20].

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