

## THE EFFECTIVENESS OF THE COORDINATION COMPOUNDS TO IMPROVE THE RHEOLOGICAL PROPERTIES OF OILS DURING TRANSPORTATION

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**Abstract.** For the first time, a complex compound of zinc (II) 1, 2, 4, 5-benzenetetracarboxylic acid with a porous structure was synthesized. The identity and chemical formula of the complex compound were determined according to X-ray diffraction, elemental, IR spectroscopy and derivatographic analysis. The process of thermal decomposition of the obtained compound was also studied. It has been found that although the parameters of the unit cell of the crystal are significantly different from the known complex, it retains its layered polymeric and porous structure. Studies of the rheological properties of oil during transportation depending on the content of resinous components and the intensity of asphalt-resin-paraffin deposits (ARPD). At the same time, the technological and economic effect is achieved as a result of the complete elimination of ARPD and acceleration of the process. The results of experimental studies of low-paraffin and high-paraffin oils show that the presence of a dispersed system of asphaltenes and resins in oil can lead to depressive effects. According to the results obtained, it was found that asphaltene-resinous components are natural depressants that lower the wax crystallization temperature depending on the type of oil. In the work, it was found that the greatest decrease in oil viscosity is observed in the presence of the BAF-1 additive. The reagent we obtained was BAF-1 obtained from the newly synthesized substance. To clarify the mechanism of action of the reagents, we conducted radiographic and electron-microscopic studies.

**Keywords:** Asphalt-resin-paraffin deposits, chemical composition of oil, complex, porous, structure, decomposition, benzenetetracarboxylic, Balakhany heavy.

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### 1. Introduction

The construction of molecular supramole-kular materials that combine macroscopic pores or have a channel structure is a complex, but unusually fruitful field of research. Such materials can be used in the petrochemical industry, in ecology for cleaning the environment, for selective separation of hydrocarbon fractions, for the production of pure n-paraffins and olefins, in the treatment of hydrocarbons, for the dewaxing of oils, for the

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separation of unsaturated compounds, for the separation of substances of normal and iso-structure, for the separation of racemates and optical isomers, etc. (Ismayilov *et al.*, 2017). To date, silicas and aluminum silicates having a microporous structure, thermal stability, as well as the ability to resize framework molecules with a template synthetic approach have been widely used from the above areas (Kusi-Sarpong *et al.*, 2018).

Supramolecular compounds have polymer layered structure containing channels and macroscopic pores, having the ability to change sizes with template synthetic approach, able to form a huge number of nanostructures or supramolecular structures leading to the creation nanotechnology, nanomaterials, different unique properties and prospects of application. Coordination polymers are used for improving the rheological properties of heavy oils. It has been established that composites based on coordination polymers reduce the kinematic viscosity of commercial oils, ensure their efficient transportation to pipeline, increase oil recovery of wells, effectively wash out ARPD from cavitation zones formed in pipelines.

Unfortunately, this method does not allow the creation of more universal materials with a macroporous structure, due to the too complex nature of building blocks (Taborda *et al.*, 2017). Studies have shown that more diverse supramolecular materials containing macroscopic pores or having a channel structure can be prepared based on inorganic coordination compounds (Zhang *et al.*, 2017). When constructing nanostructured coordination compounds, the ligand plays a decisive role, since if the ligand is large enough, then large cavities will form between the compounds by metal centers, so the choice of ligand is of great importance (Anto *et al.*, 2020). One of these ligands can be polybasic aromatic acids, in particular phthalic, terephthalic and pyromethylic acids (Hasan *et al.*, 2010). These acids have sufficient length and stiffness to form polymers with channel layered structures with metals. These coordination polymer-based supramolecular structures can form a huge number of nanostructures or supramolecular structures, leading to the creation of nanotechnology, nanomaterials, marked by unique properties and application prospects (Usubaliev *et al.*, 2017).

The search for literature material on clathrate compounds based on metal complexes of dibasic aromatic acids did not give a positive result therefore, the literature contains materials on complex compounds and adducts of metal complexes with phthalic and terephthalic acids. In accordance with GOST 12.1.007.76, phthalic acid is a toxic highly hazardous substance according to the degree of impact on the body - the 2-nd hazard class, terephthalic acid have low toxicity. Thus, the present study is devoted to the preparation of coordination compounds of metals with polybasic acids, in particular ortho, paraphthalic and 1,2,4,5-benzenetetracarboxylic acids (Zhu *et al.*, 2018). Coordination polymers have been used for the first time to improve the rheological properties of heavy oils. It has been found that when polymers interact with oil, completely destroying ARPD ensure their equal distribution in oil, and therefore, the rheological properties of oil are improved (Usubaliev *et al.*, 2017).

## 2. Materials and metohods

In order to obtain and structurally chemical study of complexes of some d-elements with polybasic aromatic carboxylic acids, the following were used as starting compounds: 1. benzoic acid (GOST 6413-77); 2. 1, 2 - and 1, 4 - benzene dicarboxylic acids (o - and p - phthalic acids) (GOST 4556-78; Code-13 107 2500; Cod - 180722500); 3. 1, 2, 4, 5 benzenetetracarboxylic (pyromellitic acid content BEKTON 197376 Russia);

4.  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ,  $\text{Fe}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$ ,  $\text{FeCl}_2$ ,  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ,  $\text{Zn}(\text{SN}_3\text{SOO})_2$ ,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{NaHCO}_3$  (Andersen *et al.*, 2005:19). The starting substances were  $\text{C}_6\text{H}_2(\text{COOH})_4$ ,  $\text{Zn}_2(\text{CH}_3\text{COO})_2$  of the CC qualification (GOST 3759-75). The complex was prepared by reacting pyro-mellitic acid with zinc acetate at a stoichiometric ratio of 1:2. In a slightly acidic medium (PH = 6.8), the solution was boiled until the acetic acid odor disappeared, filtered hot, and cooled to ambient temperature (Sharma *et al.*, 2019). Upon cooling, clear small single crystals fell out of the solution, which were filtered and washed several times with warm distilled water, dried on filter paper at room temperature. The elemental composition of the synthesized compound is shown in Table 1.

**Table 1.** The results of elemental analysis of the complex compounds of copper and zinc (II)

Found out, %			Composition of compounds	Calculated, %		
H	C	Zn, Cu		Zn, Cu	C	H
-	-	-	$\text{Cu}_2\text{C}_{10}\text{H}_{22}\text{O}_{18}$	22,812	21,541	3,950
2,315	26,721	29,011	$\text{Zn}_2\text{C}_{10}\text{H}_{10}\text{O}_{12}$	28,877	26,505	2,209

A large number of works are devoted to the structural study of metal complexes with benzoic acid and derivatives. As we have shown in the previous chapters, in recent years we have been intensively studying complexes with dibasic -1, 2 and 1, 4-phthalic acids due to the possibility of widespread use as molecular sieves and adsorbents due to their layered structure. In order to obtain more volumetric sorbents, it is of interest to study complexes with polybasic 1,2,4,5 benzenetetracarboxylic-pyromellitic acid (Martinez-Palou *et al.*, 2011).

Complex connection is highly crystalline and has high symmetry with X-ray diagram indication, unit cell parameters are calculated:  $a = 9.78$ ,  $b = 19.7$ ,  $C = 11.76 \text{ \AA}$ . Its decomposition begins at  $90^\circ\text{C}$  in the temperature range of the  $90\text{-}138^\circ\text{C}$  and is accompanied by a shallow but clear endothermic effect with a maximum at  $110^\circ\text{C}$  and corresponds to the removal of two water molecules. The experimental weight loss value is 8% (calculated 7.95%).

After that, a second endothermic effect immediately appears on the DTA curve in the temperature range  $138\text{-}180^\circ\text{C}$  with a maximum at  $150^\circ\text{C}$ , which corresponds to the removal of 1.5 moles of water. The experimental weight loss value is 6% (5.96% calculated). A third shallow and indistinct endothermic effect then appears in the temperature range  $180\text{-}280^\circ\text{C}$  with a maximum at  $240^\circ\text{C}$ , which corresponds to the removal of another 0.5 mol of water. In this case, the weight loss is experimentally 2% (calculated 1.99%). The anhydrous intermediate complex is stable to  $400^\circ\text{C}$ , which is extremely rare for complex compounds. When  $400^\circ\text{C}$ , at first gradually, then at a high speed, the anhydrous complex decomposes at the pace-rate interval  $400\text{-}600^\circ\text{C}$  with a single clear exothermic effect with a maximum of  $520^\circ\text{C}$ .

In this case, the weight loss is experimentally 48% (calculated 48.15%). Since there is no increase in mass on the TG curve after complete decomposition, it can be concluded that the oxidation of the cyn ion occurs due to oxygens of carboxyl groups.  $\text{ZnO}$  remains as the final product. The weight of the final product is experimentally 36% (calculated 35.95%). The main thermal data of the complex compound are shown in Table 2.

Thus, the result of the thermogravimetric study showed that the studied complex has the chemical formula  $\text{Zn}_2[\text{C}_6\text{H}_2(\text{COO})_4](\text{H}_2\text{O})_4$ , which is well consistent with the

formula obtained from the elemental analysis data. As can be seen, the four molecules included in the complex compound leave the crystal lattice in stages.

This indicates that they are connected to the central atom with different strengths, that is, M-O (H<sub>2</sub>O) has different meanings. IR spectroscopic examination also indicates that frequencies at 461; 534; 553 and 590 cm<sup>-1</sup> refer to vibrational oscillations of crystallization water or torsional oscillations of water molecules limited by interactions with adjacent atoms (Nurullayev *et al.*, 2019). In addition, absorption bands are observed in the IR spectrum of the compound at 3550-3200 cm<sup>-1</sup> (asymmetric and symmetrical valence fluctuations of the OH<sup>-</sup>) and at 1630-1600 cm<sup>-1</sup> (deformation fluctuations of the HOH), which are the nature of the crystallization water.

**Table 2.** Main thermal parameters of the complex connection

Connections	T <sup>max</sup> <sub>endo</sub> , °C	loss in weight				
					found	calculated
Zn <sub>2</sub> (C <sub>6</sub> H <sub>2</sub> (COO) <sub>4</sub> )(H <sub>2</sub> O) <sub>4</sub>	-	-	-	-	-	-
-2H <sub>2</sub> O	90-138	110	-	-	8,00	7,95
-1,5H <sub>2</sub> O	138-180	150	-	-	6,00	5,96
-0,5H <sub>2</sub> O	180-280	240	-	-	2,00	1,99
Acid anion without two oxygen atoms	-	-	400-600	520	48,00	48,15
ZnO	-	-	-	-	36,00	35,95

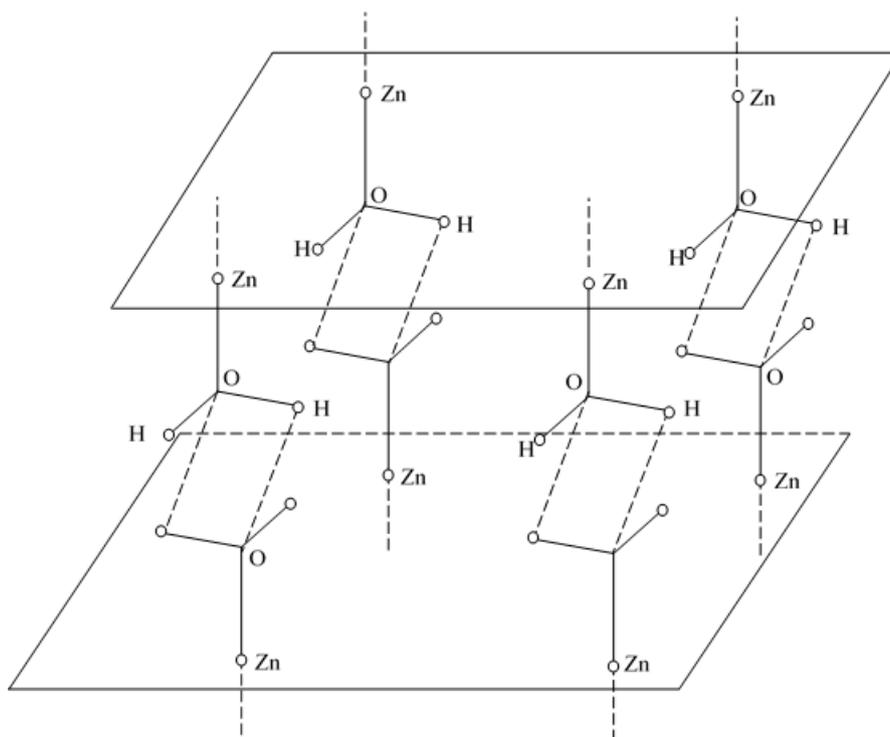
The absorption bands at 1597, 1548, 1505 (va) and 1457, 1401, 1337 cm<sup>-1</sup> (vs) are from-carried to the carboxyl groups of the acid anion. The Δ difference values (va-vs) are 140, 146 and 127 cm<sup>-1</sup>, respectively, and are significantly smaller than those of the ionic compounds, but are well matched with the values of the chelate bidentate complexes (Nurullayev *et al.*, 2016).

Thus, the central atom is sixcoordinated. The coordination of zinc (II) includes four oxygen atoms of two carbonyl groups and two oxygen atoms of water molecules. Coordination polyhedron - octahedron. The structure of the complex compound Zn<sub>2</sub>[C<sub>6</sub>H<sub>2</sub>(COO)<sub>4</sub>](H<sub>2</sub>O)<sub>4</sub> consists of cher-blowing layers along the axis (011) (Al-Adwani *et al.*, 2019). The structure is porous and the pore size is approximately 9×16Å. The layers are cross-linked by hydrogen bonds formed by coordination water molecules located in different layers at the vertices of octahedra (Fig.1).

It can also be assumed that in the structure, the framework pores will be located above each other and in this case the columns available for "guest molecules" will form.

The established structural and chemical features of the obtained compounds with phthalic, terephthalic and pyromellitic acids and their destruction can be used to realize new compounds with structures that have practically important properties. Composites based on some synthesized coordination polymers can be suitably used in the oil industry, in particular to improve oil recovery from formations and wells, to reduce the viscosity of heavy commercial oils in order to efficiently transport them through pipelines, to wash cavitation zones and ARPD formed in pipelines and tanks, respectively (Nassar *et al.*, 2010). The reliability of the results of studies on which scientific provisions and conclusions are based is based on the use of modern physicochemical methods of

elemental X-ray phase (Commander Sample ID (Coupled Two Theta/Theta WL. 1.54060), IR-spectroscopic (SPECORD-M80400-4000cm<sup>-1</sup>), thermographic (NETZSCH STA 449F3 STA449F3A-0836M), electron microscopic (Carel Zeiss Sigma brand scanning electron microscope SEM), assays and experimental methods for determining density (GOST 3900-85), kinematic viscosity (GOST 33-2000), saturated vapor pressure (GOST 1756), content of asphalt-resinous substances (GOST 11858), paraffin (GOST 11851-85 oil) and other oils and petroleum products.



**Fig. 1.** Cross-linking of layers through hydrogen bonds in the structure complex  $Zn_2(C_6H_2(COO)_4)(H_2O)_4$

### 3. Experimental part

The results of experimental investigations have shown (Table 3) that reagents BAF-1 (technical conventional name reagents) reduce the viscosity of petroleum such as heavy and crude oil (Balakhany heavy), to facilitate transportation of the subterranean formation from the production site to the refinery or oil storage tanks, increased production, purified oil and oil from the sludge in the tanks.

As is known all these properties of oils (high viscosity, difficulty of transportation, the decline in production, the sludge formation in tanks, etc.) directly related to forming asphaltene-resin-paraffin sediments ARPD (Montes *et al.*, 2019). It has been established that resinous components, which differ in composition and polarity depending on the type of oil, are natural depressants that reduce pour point of oils and petroleum products. It was noted that the presence of resins in the system can lead to both positive and negative depressant effects.

X-ray diffraction and electron microscopic studies conducted to elucidate the mechanism of action of reagents us. To conduct the study, two samples of oil (for 300

ml) from the same wells taken. To one sample of the oil added 40ml composite reactant solution, and another - left unchanged and distilled to their obtaining the fraction of tar.

As is known, tar is a dry distillate oil at 450-600 °C temperature (depending on the nature of oil) in vacuum condition and atmospheric pressure. The yield of tars depending on the oils composition is 10-45 % (wt.). Resinis a black viscous liquid and formed during the fragmentation of small glittering particles. The composition of the sludge includes paraffin, the naphthenates aromatic hydrocarbons (45-95 %), asphaltenes (3-17 %), petroleum resin (2-38 %) and the atoms of metals. Depending on the nature of the oil and from the entrance of the transparent fraction of the density of the sludge varies from 0.95 to 1.03 g/cm<sup>3</sup>, coking 8-26 % (weight) and the melting point of the 12-55 °C. As is known at atmospheric and vacuum distillation the chemical composition of the oils not change. Therefore, test results obtained for dry and oil products relates directly to oils themselves. Given the above, the dry product of oil samples subjected to X-ray diffraction and electron microscopy studies. The results are shown in Figs. 2, 3, 4 and 5, respectively. X-ray analysis and electron microscopic images obtained respectively from X-ray diffraction (Fig.2) shows that the dried product sample without reagent pronounced bright consists of three phases between planar distances 4.44, 4.22 and 3.64.

**Table 3.** Changes in the dynamic viscosity of oil extracted from the Balakhani heavy field in Azerbaijan at different temperatures

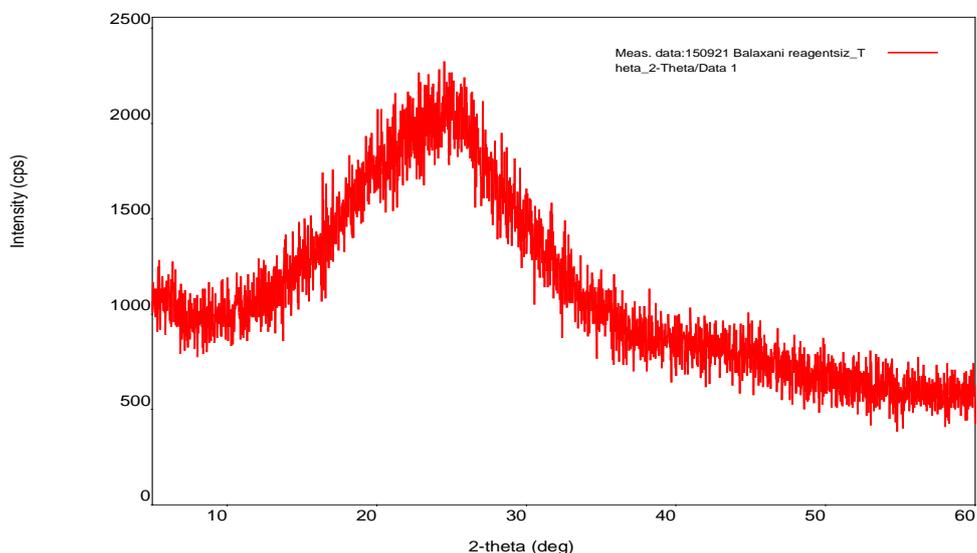
Stages of speed (corresponds to s <sup>-1</sup> velocity gradient)	Dynamic viscosity, after addition of Baf-1 reagent (0.8 kg/t) to oils, sPz .					
	20 °C		10 °C		5 °C	
	Balakhani heavy, clean	Balakhani heavy, with reagent	Balakhani heavy, clean	Balakhani heavy, with reagent	Balakhani heavy, clean	Balakhani heavy, with reagent
1a (0,3333)	681,3	596,8	996,7	748,6	1214,3	1182,36
2a (0,6)	421,9	387,6	653,2	507,4	792,7	765,9
3a (1,0)	363,5	314,9	534,6	421,9	613,9	588,2
4a (1,8)	214,8	195,3	297,8	204,1	417,2	387,9
5a (3,0)	112,3	94,8	189,3	112,6	209,6	187,4
6a (5,4)	69,7	57,4	111,8	85,9	127,4	102,5
7a (9,0)	43,9	38,4	84,5	72,7	96,3	81,7
8a (16,2)	23,8	17,4	41,2	33,7	58,9	50,2
9a (27,0)	10,3	7,9	19,9	13,5	27,4	21,3
10a (48,6)	6,1	4,6	12,4	12,4	18,2	12,8
11a (81,0)	4,3	3,5	7,5	5,1	11,4	9,7
12a (145,8)	2,8	2,3	4,9	3,7	8,1	7,4

On the radiograph (Fig. 3) are removed from the dry product oil sample with the reagent, the third phase completely disappears in the first two phases of the maxima are shifted to the low-angle side, i.e. between planar distances increase.

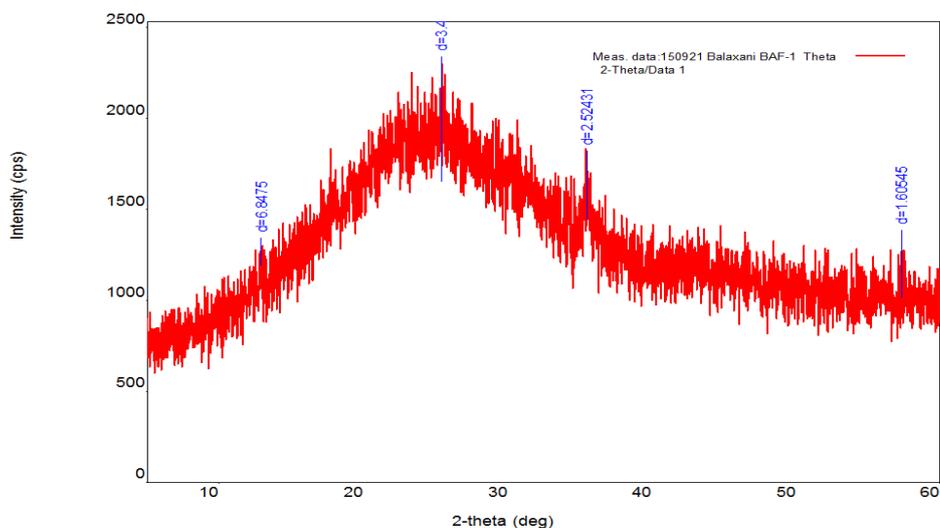
This suggests that after the disappearance of the third phase, the structure of 1-st and 2-nd phase changes and their crystallinity increases.

X-ray analysis and electron microscopic images obtained respectively From X-ray diffraction (Fig. 2) shows that the dried product sample without reagent pronounced bright consists of three phases between planar distances 4.44, 4.22 and 3.64. On the radiograph (Fig. 3) are removed from the dry product oil sample with the reagent, the

third phase completely disappears in the first two phases of the maxima are shifted to the low-angle side, i.e. between planar distances increase. This suggests that after the disappearance of the third phase, the structure of 1-st and 2-nd phase changes and their crystallinity increases.

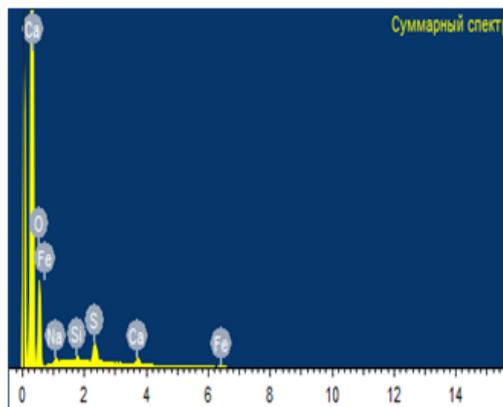


**Fig. 2.** Radiograph of a dry oil sample without reagent



**Fig. 3.** Radiograph of a dry sample oil with reagent

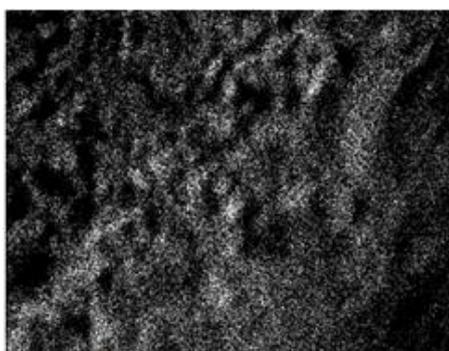
The results of electron microscopic studies reagent less sample (Fig. 4) showed that in the dry rest there is the associate consisting of asphaltenes, resins and waxes that worsen the rheological properties of crude oils.



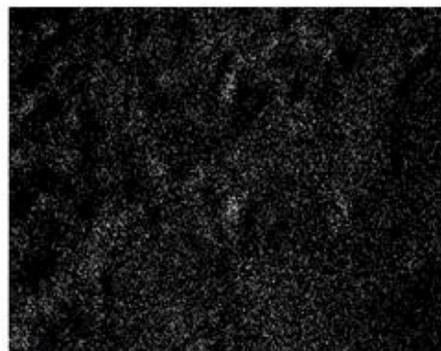
Na K	8.19	7.84	11.04	Na <sub>2</sub> O
Si K	3.82	2.99	8.18	SiO <sub>2</sub>
S K	22.35	15.34	55.81	SO <sub>3</sub>
Ca K	10.83	5.95	15.16	CaO
Fe K	7.65	3.01	9.82	FeO
O	47.17	64.88		
Итого	100.00			



Fe Ka1



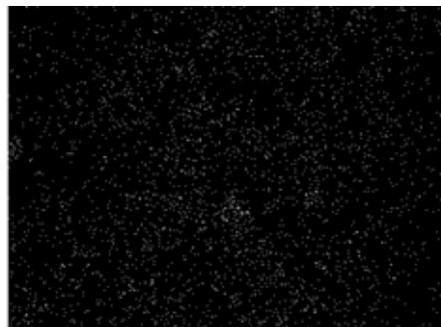
C Ka1\_2



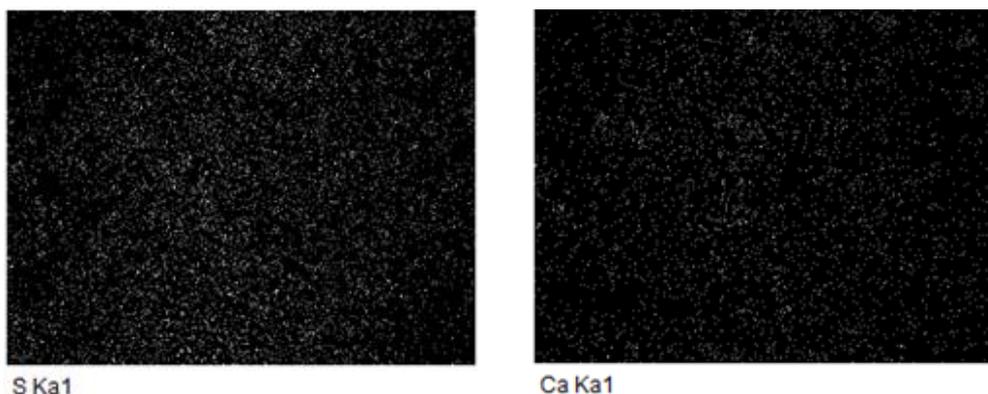
O Ka1



Na Ka1\_2

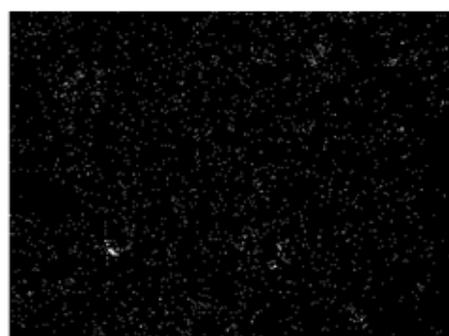
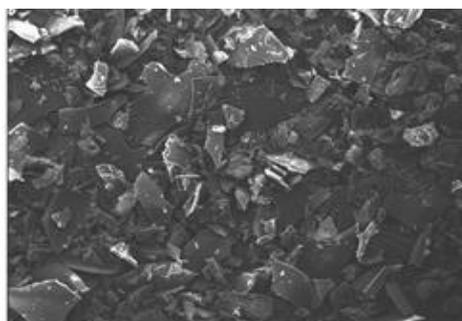
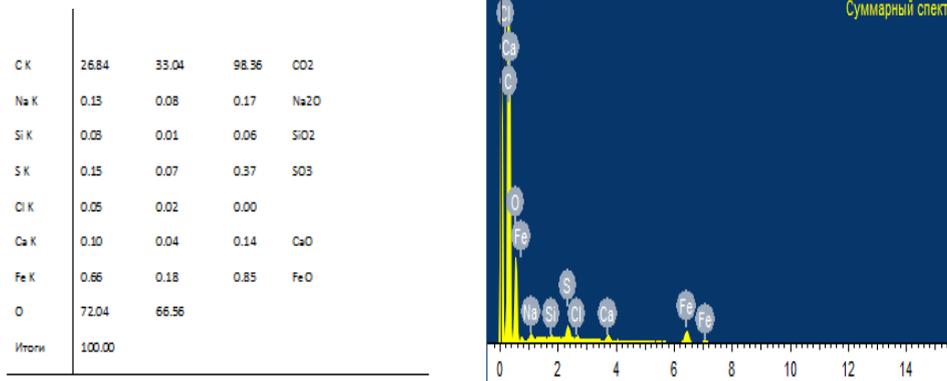


Si Ka1



**Fig. 4.** Electron- microscopic image of a dry oil sample without reagent

The results of electron-microscopic investigations of the sample with the reagent (Fig. 5) indicate that in this associate is fragmented and are distributed evenly in oil, which is in a dissolved state.



O Ka1

Na Ka1\_2

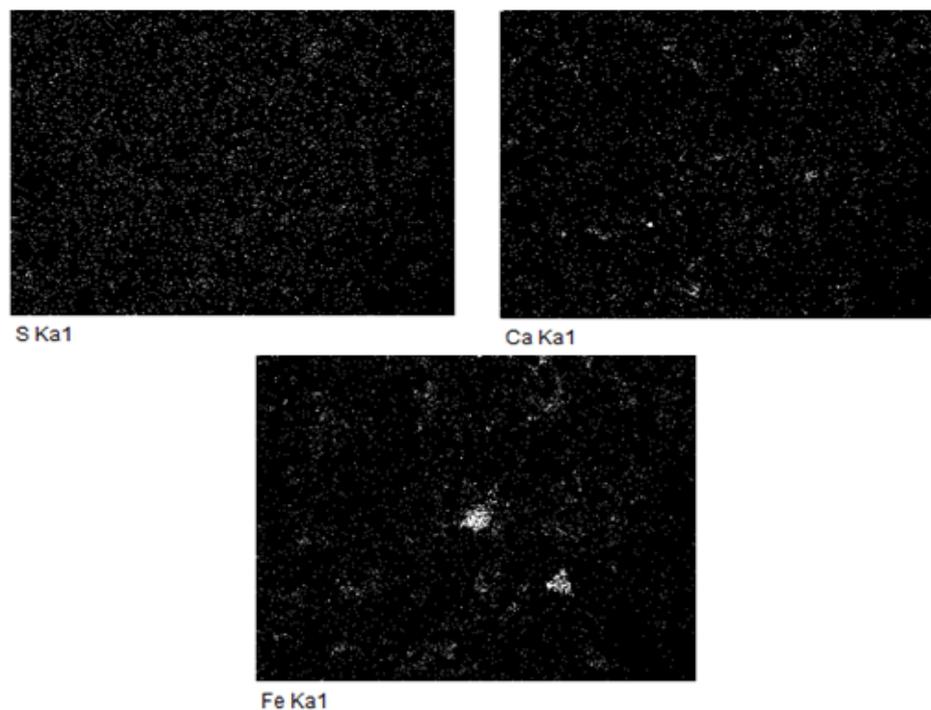


Fig. 5. Electron microscopic image of a dry sample reagent free oil with reagent

#### 4. Results and discussion

The purpose of this work is to study the mechanism of action of a multifunctional reagent on oil, which we have obtained for the first time on the basis of a coordination nanostructured polymer. The disappearance of the third phase with between planar distance ( $d = 3,60$ ) reveals that the reactants porous (pore size is  $20\text{Å}$ ) BAF-1 with the third phase non-valence form compounds with self-organization and self-construction, i.e. by reacting a reagent with oil arise non-valence skeking interaction between porous polymers and chromatic focal and heteroatom makroassociates third phase. liberated from the third phase of the first and second associates ARPD turn into small particles and dissolve in the crude oil. In this regard, and improved rheological properties of oil. An increase in the between planar spacing of the first and second phases shows that their structure changes. First synthesized and carried out systematic structural-chemical studies of supramolecular compounds of metals with polybasic aromatic carboxylic acids, such as phthalic, terephthalic and pyromellitic. At as a result of the research, it was found that all synthesized compounds have a polymer-layered structure. Coordination polymers were first used for improving the rheological properties of heavy oils. established, that polymers, when interacting with oil, completely destroying ARPD ensure their equal distribution in oil, in connection with which, improve the rheological properties of oil. Thus, the research results show that these reagents can solve all the problems that arise with ARPD, in particular, they improve the oil recovery of wells by reducing the viscosity of heavy oils, increase the efficiency of transporting such oils and ensure the efficiency of cleaning oil reservoirs from oil deposits and oil products, that is, the technology based on these reagents is multifunctional.

### 3. Conclusion

According to the results of electron microscopic analysis also clear that in the dry product without reagent paraffin oil associates are in a solid mass, which deteriorate its properties. A dry product oil with a reagent ARPD associates fragmented into small particles, i.e. they uniformly distributed in the oil and therefore improves the rheological properties of oil. This again proves that the reactants form a hetero chromatic non valence compound.

Thus, the research results show that the reagents can solve all the problems arising from paraffin formation, particularly, improve the recovery of oil wells, reducing the viscosity of heavy oils, improve the efficiency of transportation of such crudes and perform effective cleaning oil tanks of oil and petroleum deposits, then is based on the technology of said reactant is multifunctional.

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