ISSN: 2277-9655
Impact Factor: 4.116
CODEN: IJESS7

IJESRT

INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY

IR SPECTRAL AND ELEMENTAL RESEARCH SCALE INHIBITOR BASED ON ZN-HEDP

H.I. Kadirov*, O.A. Salihova
* Tashkent Institute of Chemical Technology, Uzbekistan

DOI: 10.5281/zenodo.322493

ABSTRACT

Carried out targeted studies on the development and research of properties Zn-HEDP. Studied the infrared and X-ray photoelectron spectra obtained Zn-HEDP and scale inhibitors based on them. By IR spectroscopy coordination PO3 group Zn atom is conserved localized π-connection P = O, oxygen atoms are not equal PO3 group.

KEYWORDS: organophosphonates, scale inhibitors, zincate hydroxyethylidenediphosphonic acid, bottoms residue of monoethanolamine, IR-spectroscopy, stretching vibration, bending vibration, a covalent bond.

INTRODUCTION

The problem of protecting equipment of process from scaling and internal corrosion always remains relevant in the heating engineer. One of the most effective approaches to its solution is the use of scale and corrosion inhibitors.

The most effective inhibitors of scaling and corrosion are the organophosphonic acid complexes with transition metals, among which the most practical use is obtained, as already mentioned, higher complexes hydroxyethylidenediphosphonic acid (HEDP) and zinc (zincate-HEDP), stable in the form of sodium or potassium salts. Modern scientific research [1,2] exhaustively established the mechanism of action of these inhibitors.

OBJECTS OF RESEARCH

The object of research are HEDP, HEDP-zincate, monoethanolamine and an inhibitor of deposition of mineral salts obtained with the addition of a vacuum distillation bottoms monoethanolamine.

Research methods. IR spectra HEDP-zincate were removed on the device «Agilent Technology FTIR-640» under the conditions of analysis: registration range 4000-400 cm^{-1}, scan number - 12.

The discussion of the results. As mentioned higher, the active components of these inhibitors are Zn-HEDP. The literature is very little information on the study of molecular structure organophosphonate zinc complexes. The lack of reliable information about the molecular structure of metal organophosphonate inhibitors prevents mechanisms understanding of their inhibitory effect. At the same time, in some studies [3,4] effective methods of investigation is considered IR spectroscopy and X-Ray, which allows you to explore the structure of electron-energetic atom-molecular systems, including electrons and core-valence levels.

Contact, in the wound, provides methods for preparing Zn-HEDP and mineral salt deposition inhibitors (conventional name IOMS-extra-2.1) on their base in the presence of glycerol [5]. It should be noted that the inhibitory effectiveness of IOMS-extra-2.1 in waters with different hardness is not less than 90.0 % and the inhibitor is recommended for testing under industrial conditions in water circulation systems. In order to prove the formation of zincate-HEDP and IR spectra (Fig. 1) were taken to compare the structure of the inhibitor based on it.

ISSN: 2277-9655
Impact Factor: 4.116
CODEN: IJESS7

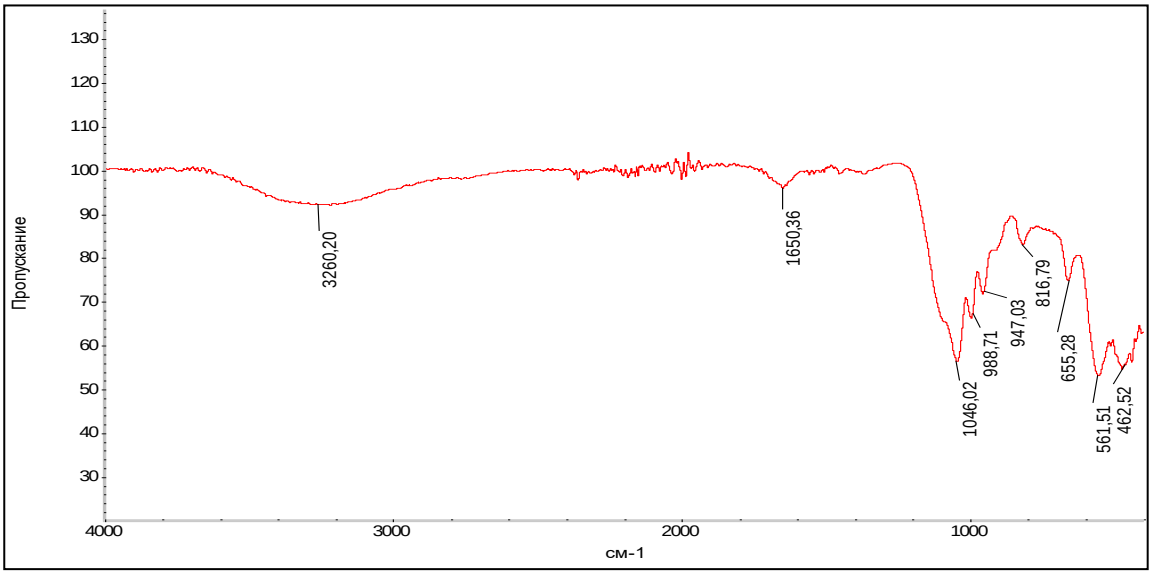


Fig. 1. IR spectra of Zn-HEDP

The spectra of the compound are present for valence vibrations of C-H bonds in the field 3400-3200 cm^{-1}; band at 1650-1640 cm^{-1} related to the localized communication P = O; band at 1060-1050 cm^{-1} related to the symmetric stretching vibrations group PO_2; band at 1000-990 cm^{-1} refers to the stretching vibrations of the group entirely deprotonated PO_3; They are also present in the band 960 - 940 and 990 - 980 cm^{-1}, which belong to the stretching vibrations of the P-O (H) of protonated phosphate groups, suggesting that the complexes are partially protonated; band at 840-820 cm^{-1} to the stretching vibrations of the C-C; band at 670-640 cm^{-1} refers to the stretching vibrations of the C-P, an intense band at 570-550 cm^{-1} refers to the stretching vibrations of the Zn-O; The absorption band at 480 - 460 cm^{-1} related to the deformation vibrations of -O-P-O.

Monoethanolamine (MEA) is a large-product of the chemical industry. MEA is found in the composition of KOMEI, and therefore of interest to study the infrared spectra obtained zincate complexes HEDP in the presence of this reagent.

IR spectra shows that absorption band at 1680 cm^{-1} refers to deformation vibrations of the water present in the product; weak bending vibrations N - H bonds in areas 1660 - 1650 and 850 - 800 cm^{-1} bending vibrations band at 1600 - 1460 cm^{-1} attributable to NH_2 group in the 1460-1440 cm^{-1} strong narrow absorption band to - CH_2- group; symmetric and asymmetric stretching vibrations group PO_2 band at 1040 - 1030 cm^{-1}; absorption bands of P - N in the 1110 - 930 cm^{-1}; bands at 910 - 900 and 970 - 960 cm^{-1} are to power fluctuations of the P-O; band at 660 - 650 cm^{-1} related to the stretching vibrations of the C - P, at 810 - 800 cm^{-1} to the stretching vibrations of the C - C; intensiveness band at 580 - 560 cm^{-1} related to the stretching vibrations of the Zn-O; absorption band at 490 - 480 and 450 cm^{-1} bending vibrations are -O-P-O.

ISSN: 2277-9655
Impact Factor: 4.116
CODEN: IJESS7

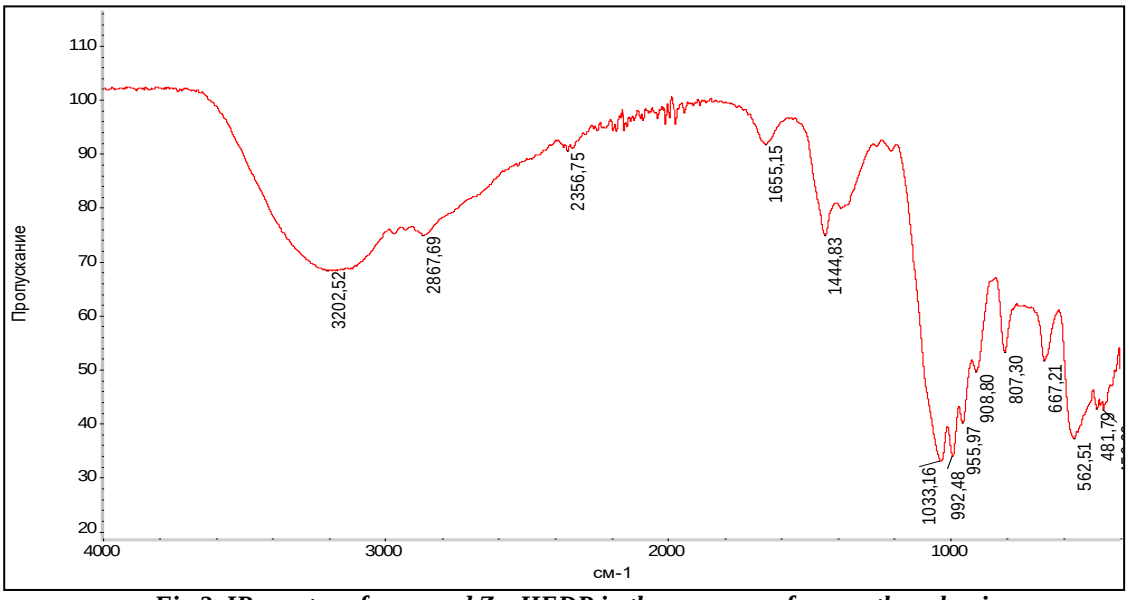


Fig.2. IR spectra of prepared Zn-HEDP in the presence of monoethanolamine

Particularly important may be deemed existents this IR spectra of characteristic groups NH_2^+ and P - N bonds than proved flow Zn-HEDP reaction with monoethanolamine with partial formation of phospho-azo compounds and ammonium salts zincate complexes HEDP, which is important for the efficiency of inhibition of scaling and corrosion of film-forming inhibitors.

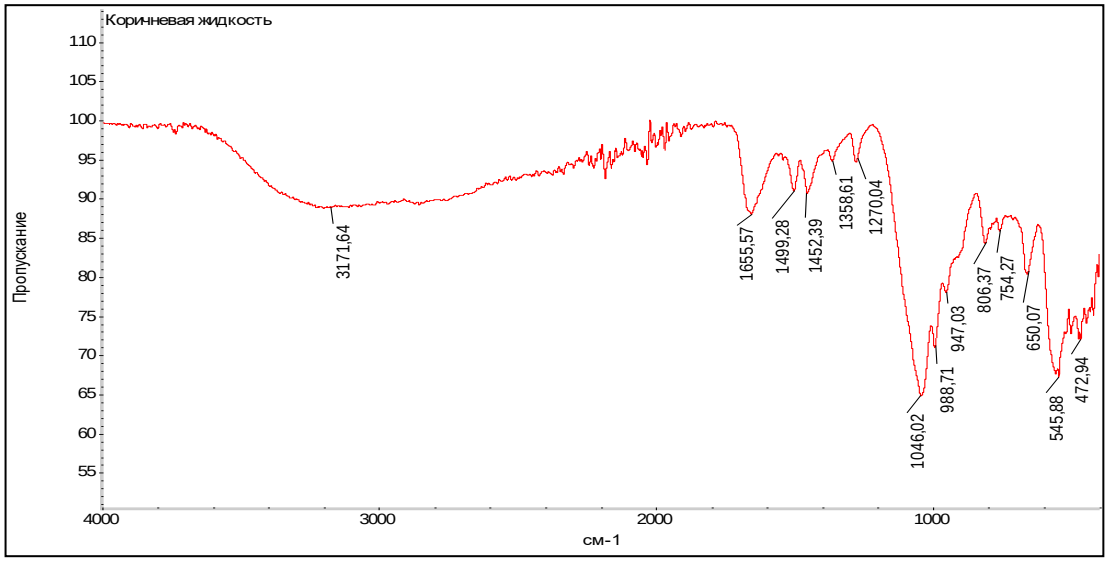


Fig.3. IR spectra of the reaction product zincate-HEDP in presence of KOMEI

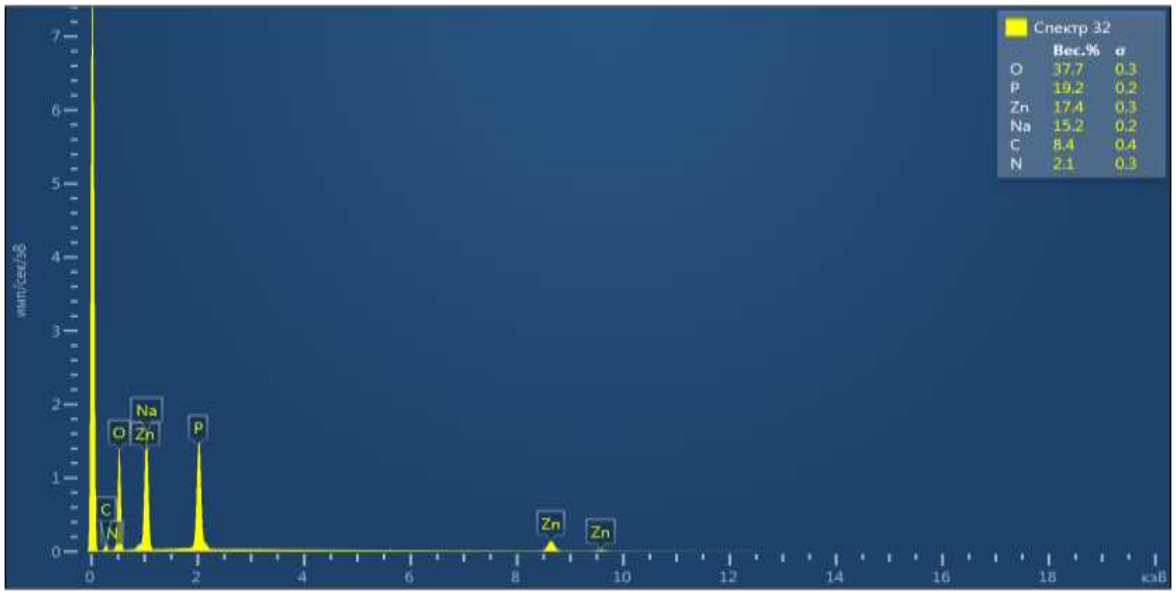
Further analyzed IR spectrum of the product-forming reaction HEDP zincate in the presence of a vacuum distillation bottoms monoethanolamine.

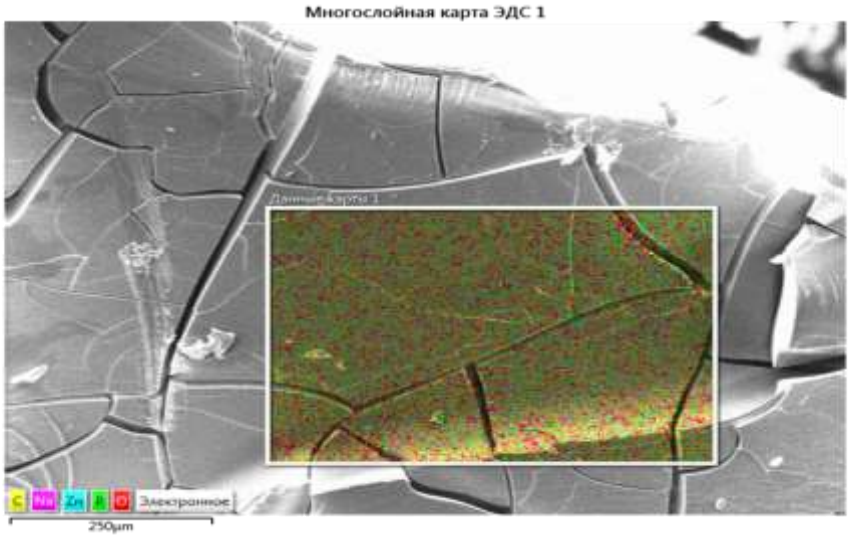
The IR spectra of the obtained product with identical IR spectra obtained Zn-HEDP complexes and IR spectra Zn-HEDP prepared in the presence of monoethanolamine. In addition, the IR spectrum there are bands bending vibrations at 1600 - 1460 cm^{-1} attributable to the NH_3^+, NH_2^+, NH^+ groups; for stretching vibrations in the 1400 - 1000 cm^{-1} N - C bond, which proves the presence of ammonium salt in the composition of the product obtained.

For elemental analysis of the crystals of the synthesized product was produced tablet (diameter 5 mm) by a hand press. The analysis was performed by scanning electron microscopy (Carl Zeiss, Germany) with energy-dispersive

ISSN: 2277-9655
Impact Factor: 4.116
CODEN: IJESS7

elemental analyzer (Oxford Instruments, UK):





Preparation of samples for analysis: Zn-HEDP selected in a sample of the heat-resistant flask, added 10 ml of concentrated nitric acid and decomposed by heating on a hot plate to obtain a clear solution. The resulting solution was quantitatively transferred to a volumetric flask of 100 ml. Thus prepared sample was analyzed with a mass spectrometer, an inductively coupled plasma in «Semiguant» mode method «TEST.M». Device parameters: plasma power of 1200 W, the integration time of 0.1 seconds.

Crystals of Zn-OEDF consist of a combination of crystals of various sizes, a few electron microscopic images taken to compile a more objective characteristics of the product under test. Even individual single crystals strongly deviate from the ideal order of strict periodicity. In the crystal lattice observed many types of violation of the order of arrangement of the atoms due to foreign inclusions, deformations, gaps (voids), deviations from stoichiometry. All this affects the properties of the surface, always having geometric and topographical complexity and heterogeneity of the force field, it will settle the existence of Zn-OEDF molecular structure with intermolecular (I) and the intramolecular (II) connection that repeatedly mentioned in the works of F.F. Chausov et al. [6-8]. These phenomena are confirmed by electron micrographs.

ISSN: 2277-9655
Impact Factor: 4.116
CODEN: IJESS7

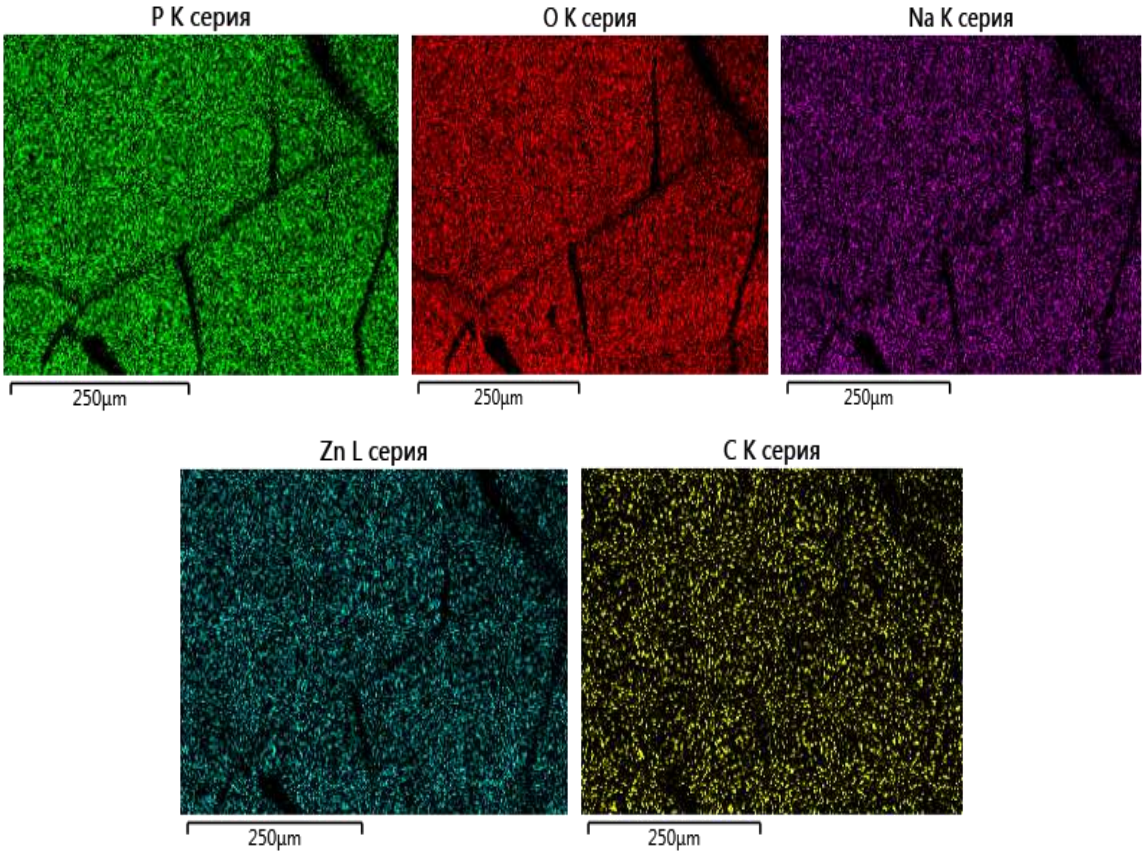
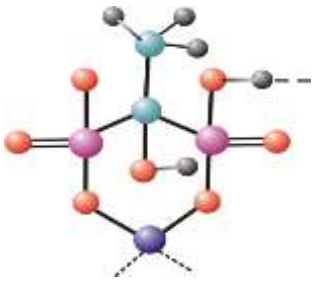


Fig. 4. Electron micrographs of the crystals obtained Zn-OEDF



Models Zn-OEDF molecular structure with intermolecular bonds

Models Zn-OEDF molecular structure with intramolecular bonds

CONCLUSION

IR spectra of revealed that the group PO_3-Zn coordination atom is conserved localized π-P = O linkages, oxygen atoms are not equal PO_3 group.

ISSN: 2277-9655
Impact Factor: 4.116
CODEN: IJESS7

REFERENCES

[1] Freiner WW (2000), 'Review of Green Chemistry Corrosion Inhibitors for Aqueous Systems' Proceedings of the 9th European Symposium on Corrosion Inhibitors, Universidad de Ferrara (Italy), Vol.1, pp. 1–37.

[2] Scale inhidition adentand method for using some: Patent 6162391 USA, IPC[7] $CO_2F5/10$, 09K 3/00. Kuritf Water Ind, Kowata Ken Ji, Limura Akira, Sato Shideru, Usui Urara, Sanaduchi Aya. № 09/083007; Stated. 21.05.1998; Pub. 19.12.2000; Pryor.23.05.1997, №9 – 150367 (Japan); NPK 422/16 Eng.

[3] Zhon Zuo-mind, Jind Guan-hua, Tong Shou-yin, Dai Uon-Zhi. Jindxi huadond zhondjianti. Making scale inhibitors adapted to the high hardness and high alkalinity. // Fine chem. Intermediates. 2001. № 4. C.21 – 24.

[4] Chemistry of Organophosphonate Scale Growth Inhibitors: 3. Vol. 3, Nos. 3-4, 2005. C.135-149. Physicochemical Aspects of 2-phosphonobutane-l,2,4-Tricarboxylate (PBTC) and Its Effect on $CaCO_3$ Crystal Growth Konstantinos D. Dcmadis and Panos Lykoudis Department of Chemistry, University of Crete, 300 Leoforos Knossos, Heraklion, Crete, Greece GR-71409.

[5] X.E. Kadirov, Z.Sh. Vohidov, T. Rizaev, U.R. Azamatov, A. Ikramov. Comparative tests organophosphonate scale inhibitors. RAS RUz. 2015. №3. p.48-51.

[6] Pat. RF. 107048. Stepanova A.G., Andreva I.R., Tarasov S.R., Filippov V.M., Ivanov V.A. A method of obtaining an aqueous solution of the disodium or dipotassium salts of zinc complex of hydroxyethylidenediphosphonic acid with a concentration of 15-23%. Stat. 06.03.2006. Опубл. 27.08.2007.

[7] E. A. Naymushina, F. F. Chausov, I. S. Kazantseva, I. N. Shabanova. Interaction of a corrosion inhibitor Zn-HEDP with carbon steel surface. Proceeding of RAS. : Physics. vol. 77, № 3, 2013, C. 362-364.

[8] I.N. Shabanova, F.F. Chausov, Y.A. Naymushina, I.S. Kazantseva. Application of X-ray photoelectron spectroscopy to study the molecular structure of the corrosion inhibitor - zinc complex of 1-hydroxyethylidenediphosphonic acid. Journal of Structural Chemistry. 2011. vol. 52. C.113-118.