Synthesis And Investigation Of New Sulphadimezine Compounds Based On α-Chloroalkyl And Acyloxymethyl Ether

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Abstract. New sulphadimidinecompounds were prepared based on ethers of α-chloroctoxymethyle, α- chlordesoxymethyle, α-chlordedesoxymetil, 2-chloro-5-oxohexen-2. As the prepared compounds all have –CH_2OR functional group in their composition the higher ecological inhibition effect of each of the said compounds were determined at 2.5, 5 and 10 mg/l concentrations.

We have already conducted some researches on organic materials containing nitrogen and sulfur and determined their effects as corrosion inhibitors. The results of our researches indicated that the organic compounds containing nitrogen and sulfur with some –CH_2OR functional group in their composition positively affects the corrosion strength of "Cm.3" steel in lands with high corrosive effect and promotes the strength of the steel against aggressive materials up to 96.7 – 100% even at lower concentrations like 2.5, 10 and 10 mg/l [1-3].

The results of scientific studies conducted on environment indicate that one of the problems that the oil and gas industries are facing with is corrosion of steel equipment used in this sector. The corrosion eventually results in explosion and fire accidents in the oil facilities which in turn cause leakage and spillage of oil and oil products into the environment. Due to this reason, the use of compounds with even 35 -50% inhibitory efficiency has been evaluated from economical point of view.

The main part of compounds, used previously, with even 35-50% inhibitory efficiency, have themselves categorized as environmentally harmful materials. Furthermore, due to their effect on the quality of the produced products and their harmful effect on labor health even intoxication caused the stop of utilization of such materials.

Recently very actual and important studies have been conducted on low-cost organic compounds capable to meet all environmental requirements with higher boiling point as well as higher inhibition efficiency at very low concentration and their feasibility from economic and environmental points of view have been evaluated.

In line with the foregoing, we carried out studies on synthesis of new and low-cost compounds from alcohols produced by petrochemical and organic chemical industries such as $C_8H_{17}OH$, $C_{10}H_{21}OH$ and $C_{12}H_{25}OH$ as well as another alcohol which is in fact one of waste materials of synthetic rubber industries namely 1,3-dichlorobutane-2 $CH_3CCl{=}CH{-}CH_2OH$. The inhibitory effect of these compounds was assessed in powerful corrosive environments at 2.5, 5 and 10 mg/l concentrations.

PRACTICAL PART

The process of synthesis of new sulfadimezine has been carried out by use of the following stages.

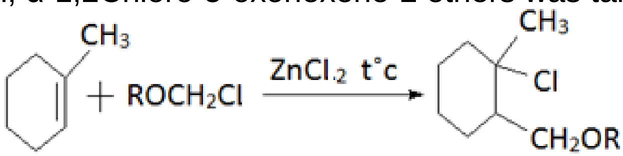
In the first stage, the α-chlorolakyl ether and alkenyloxymethyl ether were synthesized [1-2,4]:

$$CH_2O+ROH+HCl\uparrow \xrightarrow{-5^0C} ROCH_2Cl+H_2O$$

where, R=$-C_8H_{17}$;$-C_{10}H_{21}$; $-C_{12}H_{25}$; $-CH_2-CH=CCl-CH_3$

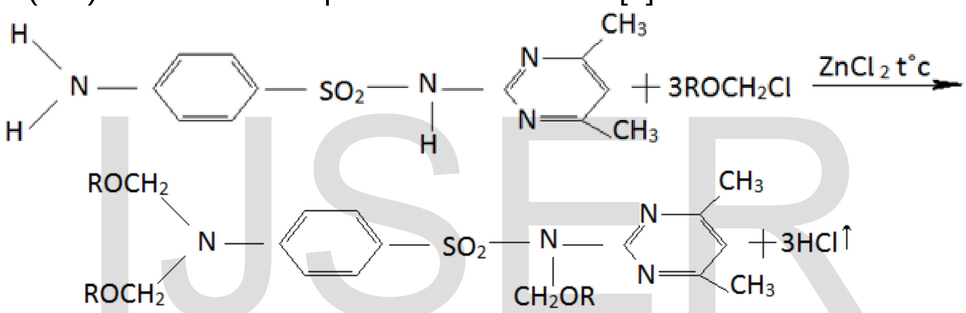
The composition, physicochemical constants, and analysis of elements of newly synthesized α-chlorolakylwere determined as per methodology and principles described in the relevant literature. The obtained results were in conformity with the information described in the relevant literature.

In the second stage, the alchoxychlororation process of 1-methylcyclohexene by α-chlorooxymethyl, α-1,2Chloro-5-oxohexene-2 ethers was taken place [2]:



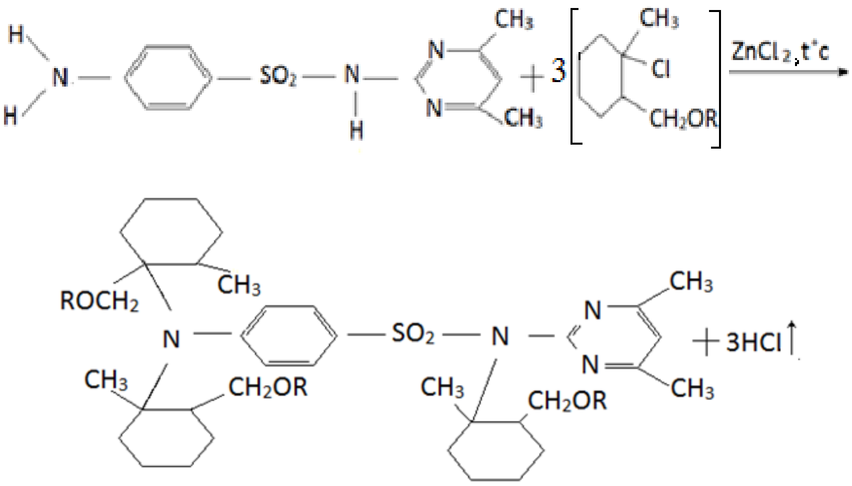
where, R= $-C_8H_{17}$; $-C_{10}H_{21}$; $-C_{12}H_{25}$; $-CH_2-CH=CCl-CH_3$

In the third stage, the reaction of sulfadimezine and α-chlor ethers was conducted (I-IV) and a new compound was obtained [2]:



where, R=$-C_8H_{17}$(I);$-C_{10}H_{21}$(II);-C12H25(III); -CH2-CH=CCl-CH3(IV).

In the fourth stage, the reaction between 1-methyl-1-chlor-2-alchoxymetyl and 1-methyl-1-chlor-2-alkeniloxy-metylcyclohexanes with sulphadimezine was conducted (V-VIII) and new compounds were synthesized [2]:



where, R=$-C_8H_{17}$(V); $-C_{10}H_{21}$(VII);$-C_{12}H_{21}$(VII); $-CH_2-CH=CCl-CH_3$(VIII)

<u>Synthesis of N_1,N_1,N-trioctyloxymethyl-sulfadimezine (I)</u>

2 g $ZnCl_2$, 27.8 g (0.1 g-mol) sulfadimezine was added to the reaction flask and 100 ml of C_2H_5OH alcohol was added to the medium. The medium was mixed at 70 °C, up to full dissolution of sulfadimezine in the alcohol. Then 53.52 g (0.3 g-mol) α-chloroctoxymethyl ether was added to the flask intermittently by use of pipette and then the medium is mixed for 8 hours at 76 °C. Then the reaction medium is neutralized by adding 100 ml of 10% NaOH in room temperature and is washed by 250 ml distilled water. The organic layer is separated by use of diethyl ether. The dissolving water is extracted by use of vacuum pump and then the organic layer is dried over $CaCl_2$.

Distillation ofsynthesized N_1,N_1,N-trioctyloxymethyl-sulfadimezine (I)compound is carried out in a vacuum apparatus

The synthesis of N_1,N_1,N-triodesoxymetiyl-sulfadimezine (II), N_1,N_1,N-trioctyloxymethyl-sulfadimezine (III), N_1,N_1,N-tri (2-chloro-5-oxohexen-2)-sulfadimezine (IV), N_1,N_1,N-tri (1-methyl-2-octoxy methylcyclohexane) sulfadimezine (V) and N_1,N_1,N-tri (methyl-2-desoxymethyl cyclohexane) (VI), N_1,N_1,N-tri {1-metyl-2-(-2-chlor-5-oxohexene-2)}-sulfadimezine (VIII) compounds was taken place.

The synthesized sulfadimezinecontent in the compound (I-VIII) was determined based on α-chloro-ethers and the composition, element structure of each of them was determined by means of IR, NMR and atomic mass spectrometry.

The intensive lines determining the specifications of groups existing in each of the compounds were determined in IR spectrometry. The measured lines intervals were as follows: C-O-C ether group: 1050, 1080 cm^{-1}, C-N bond: 1280, 1310, 1350 cm^{-1}, N-N bond: 1580 cm^{-1}, $-CH_2$ group: 2950 cm^{-1}, $-CH_3$ group: 1380, 1460, 2990,3030 cm^{-1}, $-C_6H_5$ group 700 – 780 cm^{-1}.

In IR spectrometry of IV, VIII compounds C=C bonds in 2-Cloro-5-oxohexene-2 group has been 1640 cm^{-1}, the C-Cl bond has been in 650 cm^{-1}intensity. The atomic mass spectrometry of new sulfadimezine compound (I-IV) has been conforming to 704 m/l, 788 m/l, 872 m/l and 639,5m/l. Conditionally, sulfadimezine (I-VII) in the spectrometry of newly compound NMR,H and the signals determining and characteristic to H bond in these compounds were drawn.

The information on physicochemical constants and elements of sulfadimezine synthesized based on α-chlor ethers (I-VIII) has been included in Table 1.

The study of inhibition activities of synthesized sulfadimezine in new compounds (I-VIII) was conducted by use of gravimetric analysis method and the metal mass lost.

We used 2 cm^2 of specially polished "Cm.3" steel sample in our test under laboratory conditions in powerful corrosive medium at 40 – 45 °C by use of tube with 4 outlets in a dynamic condition.

The studied inhibitor materials were applied to 1 liters of powerful corrosive media at 2.5, 5 and 10 mg/l concentrations for a period of 3 hours to determine the inhibitory effect. We used the effective inhibitor referred to in the relevant literature introduced by the fame author [5] as control.

The scientific investigations were conducted for determination of inhibitory effects of new compositions of sulfadimezine synthesized through I–VIII stages and the results have been included in Table 2.

Table 2 indicates that the effectiveness level of new compounds containing sulfadimezineeven at 2.5, 5 and 10 mg/l concentrations is very high more effective when compared with other inhibitors at several times higher concentration (200 mg/l).[6]

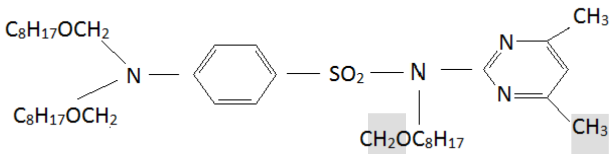
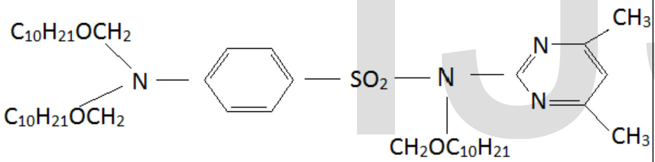
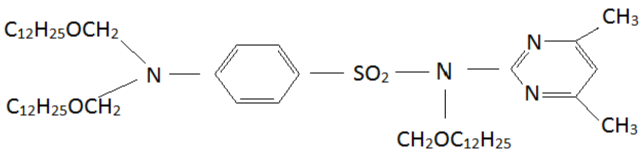
With higher probability, the new compounds containing sulfadimezine are absorbed into surface of steel sample and neutralize the activeness of corrosive material.

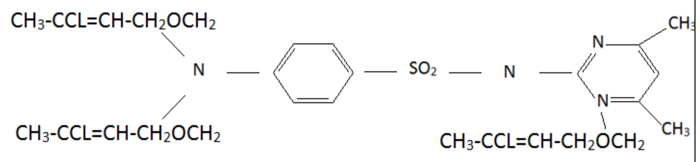
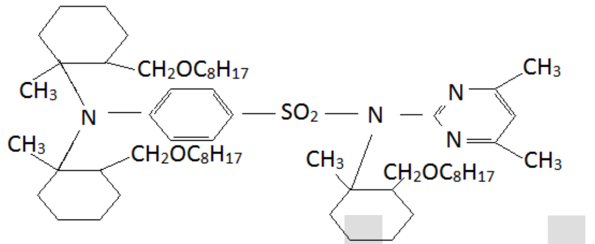
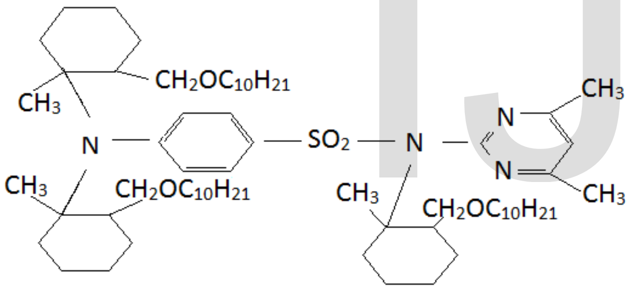
Due to double bonds between atoms of new compounds containing sulfadimezine(between HC=CH, -Ch_2OR groups) and the higher atomic density relating to the number of nitrogen in their composition neutralizes the aggressive effect of abrasive materials on steel surface. Due to complex bonds between the inhibitor compounds on steel surface, they protect the steel sample from corrosive effect of the medium.

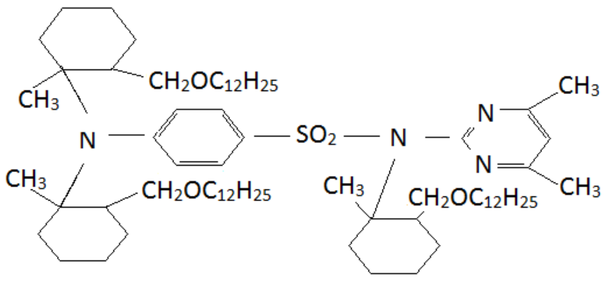
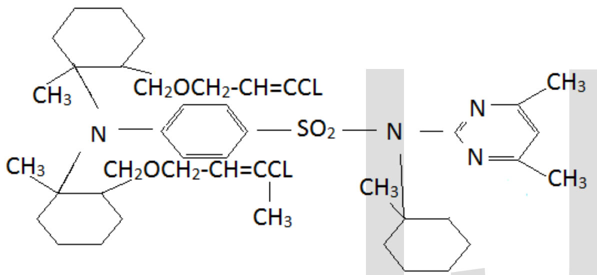
The results of scientific studies carried out by us indicate that the synthesized new compounds containing sulfadimezine protects the equipment used in oil and gas industries against powerful corrosive materials up to 250 °C; besides they're powerful corrosive inhibitors, in the same time they are environmentally clean materials and their application is justifiable from scientific point of view.

As seen from their composition, structure, new compounds containing sulfadimezine (I–VIII) could be used as medical preparations, additives, in the form of biologically active materials and we consider it necessary to conduct research in these areas.

Table 1 – Physicochemical constants and element analysis (%) of new compounds containing sulfadimezine

Chemical formula and identification number of chemicals	***Content %***	***T_{boi} ^0C (mm Hg)***	***d_4^{20}***	***n_D^{20}***	***MR_D Calculated***	***Brutto formula, mol.w***	***Calculated / found analysis of the element***				
							C	H	N	S	Cl
1	2	3	4	5	6	7	8	9	10	11	12
 I	96,85	215-216 (3)	1,0563	1,5375	208,31 / 208,12	$C_{39}H_{68}N_4O_5S$ 704	66,47 / 66,35	9,66 / 9,43	7,95 / 7,71	4,54 / 4,35	-
 II	96,79	226-227 (3)	1,0602	1,5486	236,28 / 236,0	$C_{45}H_{80}N_4O_5S$ 788	68,53 / 68,28	10,15 / 9,86	7,11 / 6,83	4,06 / 3,81	-
 III	96,76	245-246 (3)	1,0736	1,5638	264,09 / 263,18	$C_{51}H_{92}N_4O_5S$ Cl 872	70,18 / 69,85	10,55 / 10,23	6,42 / 6,18	3,67 / 3,28	-

 IV	95, 57	268- 269	1,339 7	1,59 01	161,11 160,62	$C_{27}H_{41}N_4O_5SCl_3$ 639,5	50,66 50,52	6,41 6,14	8,76 8,52	5,0 4,81	16, 35 16, 04
 V	97, 85	240- 241 (3)	1,099 2	1,57 85	299,71 299,54	$C_{60}H_{104}N_4O_5S$ 992	72,58 72,35	10,48 10,09	5,65 5,31	3,23 3,01	-
 VI	97, 63	255- 256 (3)	1,106 3	1,58 85	327,54 327,42	$C_{66}H_{116}N_4O_5S$ 1076	73,61 73,21	10,78 10,41	5,20 4,75	2,97 2,71	-

 VII	97,58	277-278 (3)	1,1184	1,6015	355,56 / 355,30	$C_{72}H_{128}N_4O_5S$ 1160	74,48 / 74,25	11,03 / 10,86	4,83 / 4,51	2,76 / 2,51	-
 X=-CH₂OCH₂-CH=CCL-CH₃ VIII	95,65	286-287 (3)	1,3502	1,6615	252,41 / 252,05	$C_{48}H_{71}N_4O_5SCl_3$ 921,5	62,51 / 62,15	7,70 / 7,36	6,08 / 5,81	3,47 / 3,12	11,56 / 11,23

СПИСОК ЛИТЕРАТУРЫ

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