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Synthesis and Characterization of New Derivatives of Trithiocarbonic Acids as Additives for Lubricating Oils

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The development of effective lubricant additives is essential for reducing friction and wear under demanding operating conditions. In this study, a series of sulphur-rich trithiocarbonate derivatives containing hydroxyl, ether, and chlorine functionalities was synthesized using epichlorohydrin-derived intermediates. The structures of the synthesized compounds were characterized by IR, ^{1}H NMR and ^{13}C NMR spectroscopy. Their tribological performance was evaluated in MC-20 transmission oil using a four-ball friction tester. The results revealed differences in lubrication performance depending on the functional groups present in the molecular structure. The hydroxyl-derivative exhibited the highest effectiveness, followed by the allyloxymethoxy- and butoxymethoxy derivatives. The improved performance of the hydroxyl containing compound may be attributed to stronger interactions with the metal surface, which can promote additive adsorption and protective boundary-film formation. The findings suggest that functional group composition plays an important role in governing the tribological behaviour of trithiocarbonate-based additives.

Keywords: Epichlorohydrin derivatives, Trithiocarbonic acids, Lubricating oil, Tribological properties.

INTRODUCTION

Wear of frictional components is a major challenge in the operation of machinery and mechanical systems as it reduces reliability increases maintenance requirements and shortens equipment service life. Strategies to minimize these effects include improvements in component design and the use of lubricants with enhanced tribological properties. The incorporation of functional additives into lubricating oils is an effective approach to reduce the friction and wear while limiting scuffing under high loads and temperatures. These additives can form protective boundary films on metal surfaces through physical adsorption and chemical interactions thereby reducing direct contact between opposing surfaces [1-6].

The selection of suitable additives is an important aspect of lubricant development. Previous studies [7-11] have established the beneficial effects of sulphur chlorine nitrogen and phosphorus-containing compounds on the anti-wear and extreme-pressure properties of lubricating oils. Trithiocarbonic acid derivatives are of particular interest due to their sulphur-rich structures, which may promote interactions with metal surfaces and facilitate the formation of protective tribofilms under frictional conditions.

Anti-wear additives reduce material loss by forming boundary films that separate contacting surfaces during sliding. Under increased frictional heating and mechanical stress these films may undergo chemical transformation or decomposition followed by the formation of more strongly bound surface layers [12-14]. The effectiveness and durability of the protective film depend on the chemical structure and polarity of the additive as well as its adsorption behaviour and reactivity towards the metal surface. Molecular interactions at the interface can support the formation of a boundary layer that limits surface damage during operation.

Anti-wear and extreme pressure additives serve complementary functions in lubricating systems. Anti-wear additives limit gradual material loss under sliding conditions whereas extreme pressure additives help prevent severe surface damage and seizure under high loads [15-18]. Their protective action

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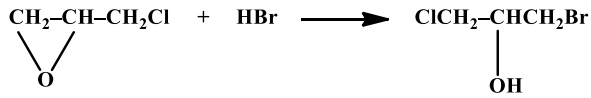
may involve reactions between active elements such as sulphur chlorine and phosphorus and the metal surface. Depending on the additive composition and operating conditions these reactions can generate protective compounds such as metal sulphides or chlorides that reduce direct metal-to-metal contact and improve load-bearing performance [2].

Based on the previous studies of trithiocarbonic acid derivatives [19,20], the present work focuses on the synthesis of a series of sulphur based compounds incorporating hydroxyl and ether functionalities. The molecular design aims to combine the surface reactivity of trithiocarbonate groups with the potential adsorption and interfacial interactions provided by oxygen containing functional groups. This study focuses on the synthesis and characterization of novel trithiocarbonic acid derivatives containing hydroxyl, chlorine and ether groups using epichlorohydrin-derived intermediates. Their structures were confirmed by IR, ^{1}H and ^{13}C NMR spectroscopy and their tribological properties were evaluated in MC-20 lubricating oil to assess the influence of functional groups on lubrication performance.

EXPERIMENTAL

All reagents and solvents were used as specified in the original experimental procedures. Infrared (IR) spectra were recorded using a Specord-75 IR spectrophotometer (Carl Zeiss, Germany). ^{1}H NMR spectra were recorded using a Bruker Fourier spectrometer (300 MHz) and ^{13}C NMR spectra were acquired at 75 MHz using a Bruker Fourier spectrometer. Tetramethylsilane (TMS) was used as the internal standard. The synthesized compounds were purified by vacuum distillation or adsorption column chromatography depending on their thermal stability. The chromatographic purification was performed using ASK-grade silica gel in a column comprising three sections with diameters of 4, 3 and 1.5 cm. Benzene/2-propanol (4:1, v/v) or chloroform/2-propanol (7:3, v/v) was used as the eluent, as appropriate. The combined eluates were concentrated under reduced pressure and the products were monitored by refractive index measurements. Product purity was determined by gas-liquid chromatography (GLC), where specified.

Synthesis of 1-bromo-3-chloropropan-2-ol: Epichlorohydrin (46.5 g, 0.50 mol) was added dropwise to 101.23 g of 46% aqueous HBr (2.20 mol). The reaction mixture was stirred at 30 ºC for 5 h and then filtered. Benzene was removed by distillation and the residue was purified by vacuum distillation to afford 1-bromo-3-chloropropan-2-ol as a product with 99% purity by GLC (**Scheme-I**). Yield: 82%, b.p.: 93 ºC at 1.2 mmHg; Density (at 20 ºC): 1.7643 g/cm^3; refractive index (n_D): 1.5183; molar refraction (MR_D, g/mol): calcd. (found):



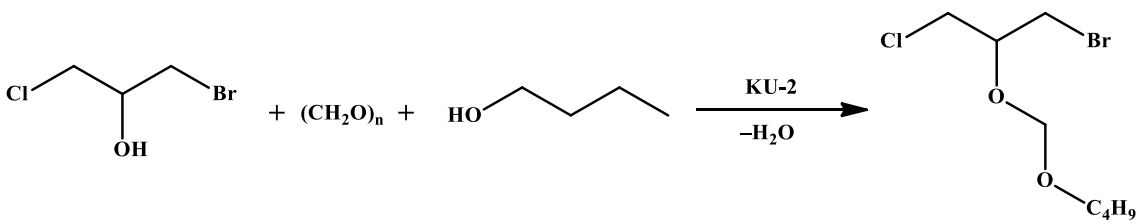
Scheme-I

30.21 (29.78); Elemental analysis of C_3H_6BrClO: calcd. (found) %: C, 20.76 (20.90); H, 3.48 (3.62); Br, 46.05 (46.20); Cl, 20.46 (20.51); ^{1}H NMR (300 MHz, acetone, δ ppm): 3.59 (m, 2H, CH_2Br), 3.70 (d, 2H, CH_3Cl), 4.07 (m, 1H, CH), 4.77 (s, 1H, OH). ^{13}C NMR (75 MHz, acetone, δ ppm): 29.24, 34.99, 46.69, 70.38.

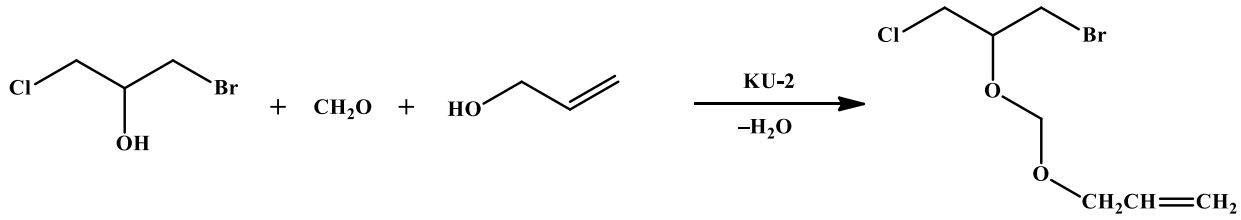
Synthesis of 1-bromo-2-butoxymethoxy-3-chloropropane: Butanol (14.82 g, 0.20 mol), paraformaldehyde (6.0 g, 0.20 mol) and 1-bromo-3-chloropropan-2-ol (37.04 g, 0.17 mol) were heated in benzene in the presence of KU-2 cation-exchange resin (10% as reported relative to the acid). The reaction was continued until the excess water had been removed by azeotropic distillation. The mixture was filtered and benzene was removed by distillation (**Scheme-II**). The residue was purified by vacuum distillation at 90 ºC and 1.2 mmHg to obtain 1-bromo-2-butoxymethoxy-3-chloropropane in 65% yield with 95% purity by GLC. Density (at 20 ºC): 1.3090 g/cm^3; refractive index, (n_D), 1.4666; molar refraction (MR_D, g/mol): calcd. (found): 55.06 (54.97); Elemental analysis of $C_8H_{16}BrClO_2$: calcd. (found) %: C, 37.01 (37.31); H, 6.21 (6.41); Br, 30.77 (30.91); Cl, 13.67 (13.81); IR (KBr, ν_{max}, cm^{-1}): 1114.70, 1041.99 (C–O–C *str.*), 755.33 (C–Br), 669 (C–Cl).

Synthesis of 1-bromo-2-(allyloxymethoxy)-3-chloropropane: Allyl alcohol (11.61 g, 0.20 mol), paraformaldehyde (6.0 g, 0.20 mol) and 1-bromo-3-chloropropan-2-ol (29.48 g, 0.17 mol) were heated in benzene in the presence of KU-2 cation-exchange resin (10%, as reported relative to acid). The reaction was continued until the excess amount of water had been removed by azeotropic distillation. The mixture was filtered and benzene was removed (**Scheme-III**). The residue was purified by vacuum distillation at 90 ºC and 1.2 mmHg to afford the product in 50% yield with 95% purity. Density (at 20 ºC): 1.4005 g/cm^3; refractive index, (n_D), 1.4810; molar refraction (MR_D, g/mol): calcd. (found): 49.97 (49.48); Elemental analysis of $C_7H_{12}BrClO_2$: calcd. (found) %: C, 34.51 (34.00); H, 4.96 (4.90); Br, 32.80 (32.21); Cl, 14.57 (14.10); IR (KBr, ν_{max}, cm^{-1}): 3081 (=C–H *str.*), 1647.59 (C=C *str.*), 1173, 1108 (C–O–C *str.*), 755.78 (C–Cl), 670.91 (C–Br), 2284-1689 (alkene-associated overtone bands).

Synthesis of butyl and pentyl trithiocarbonate salts: Butyl and pentyl trithiocarbonate salts were prepared by reacting the corresponding thiol compounds with carbon disulfide in the presence of potassium hydroxide following the reported



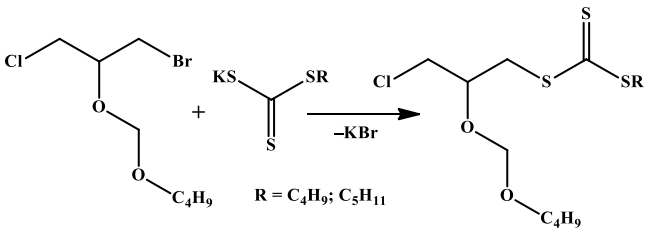
Scheme-II



Scheme-III

procedure [21]. The resulting salts were used as nucleophilic reagents in the synthesis of the target trithiocarbonate derivatives.

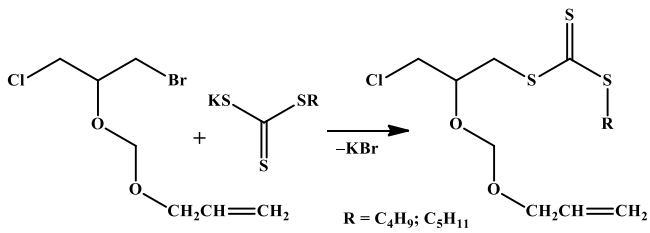
Synthesis of 2-butoxymethoxy-3-chloropropyl pentyl carbonotrithioate: 1-Bromo-2-butoxymethoxy-3-chloropropane (25.9 g, 0.10 mol) was added to a solution of potassium pentyl trithiocarbonate (26.20 g, 0.12 mol) in acetone (70 mL). The reaction mixture was stirred at 25-30 ºC for 5 h, diluted with hexane, washed with distilled water and filtered (**Scheme-IV**). Hexane was removed by distillation and the residue was purified by column chromatography using a benzene/ 2-propanol mixture as the eluent. Yield: 76%; Density (at 20 ºC): 1.1535 g/cm^3; refractive index, (n_D), 1.5582; molar refraction (MR_D, g/mol): calcd. (found): 100.80 (100.35); Elemental analysis of $C_{14}H_{27}ClO_2S_3$: calcd. (found) %: C, 46.83 (47.00); H, 7.57 (7.11); S, 26.78 (26.30); Cl, 9.88 (9.61); ^{1}H NMR: (300 MHz, $CDCl_3$-d_6, δ ppm): 0.73 (m, 6H, CH_3), 1.05-1.51 (m, 10H, CH_2), 2.71-3.69 (m, 8H, CH_2), 3.90 (m, 1H, CH), 4.50 (s, 2H, OCH_2O); ^{13}C NMR (75 MHz, $CDCl_3$-d_6, δ ppm): 13.75, 13.82 (CH_3), 19.36, 22.15, 27.77, 30.93, 31.70, 36.61, 37.07, 45.79 (CH_2), 68.06 (OCH_2), 74.56 (O–CH), 94.60 (OCH_2O), 223.49 (C=S).



Scheme-IV

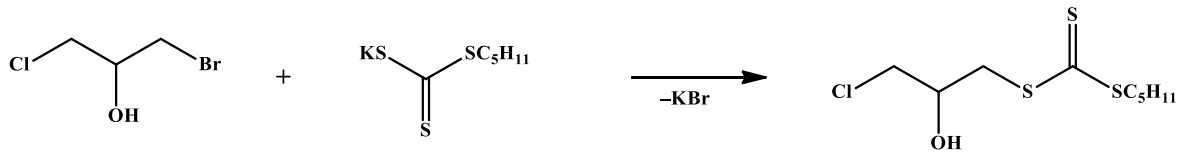
Synthesis of 2-(allyloxymethoxy)-3-chloropropyl pentyl carbonotrithioate: 1-Bromo-2-(allyloxymethoxy)-3-chloropropane (24.3 g, 0.10 mol) was added to potassium pentyl trithiocarbonate (26.20 g, 0.12 mol) dissolved in acetone (70 mL). The reaction mixture was stirred at 25-30 ºC for 5 h and then diluted with hexane, washed with distilled water and filtered (**Scheme-V**). Following removal of hexane by distillation, the crude product was purified by column chromatography using benzene/2-propanol as the eluent. Yield: 81%; Density (at 20 ºC): 1.1667 g/cm^3; refractive index, (n_D), 1.5666; molar refraction (MR_D, g/mol): calcd. (found): 95.74 (95.84); Elemental analysis of $C_{13}H_{23}ClO_2S_3$: calcd. (found) %: C, 45.52 (45.17); H, 6.75 (6.21); S, 28.04 (27.71); Cl, 10.34 (10.00); ^{1}H NMR (300 MHz, $CDCl_3$-d_6, δ ppm): 0.72 (3H, CH_3), 1.01-1.61 (m, 6H, CH_2), 3.12 (2H, SCH_2), 3.35 (2H, SCH_2), 3.55 (CH_2Cl), 3.91 (OCH), 3.92 (OCH_2), 4.50 (OCH_2O), 5.00, 5.18 (CH_2=), 5.73 (CH=); ^{13}C NMR (75 MHz, $CDCl_3$-d_6, δ ppm): 13.75 (CH_3), 22.14, 27.63, 30.88, 36.98, 38.46, 45.83 (CH_2), 68.41 (OCH_2), 74.72 (O–CH), 93.73 (OCH_2O), 116.37 (=CH_2), 134.19 (=CH), 223.46 (C=S).

Synthesis of 2-(allyloxymethoxy)-3-chloropropyl butyl carbonotrithioate: 1-Bromo-2-(allyloxymethoxy)-3-chloropropane (24.3 g, 0.10 mol) was added to a solution of potassium butyl trithiocarbonate (24.51 g, 0.12 mol) in acetone (70 mL). The reaction mixture was stirred at 25-30 °C for 5 h, diluted with hexane, washed with water and filtered (**Scheme-V**). Hexane was removed by distillation and the residue was purified by column chromatography using benzene/2-propanol as eluent. Yield: 84%; Density (at 20 ºC): 1.1843 g/cm^3; refractive index, (n_D), 1.5707; molar refraction (MR_D, g/mol): calcd. (found): 91.12 (91.44); Elemental analysis of $C_{12}H_{21}ClO_2S_3$: calcd. (found) %: C, 43.81 (44.00); H, 6.43 (6.21); S, 29.23 (29.00); Cl, 10.79 (10.81); ^{1}H NMR: (300 MHz, $CDCl_3$-d_6, δ ppm): 0.70 (3H, CH_3), 1.01-1.60 (m, 4H, CH_2), 3.12 (2H, SCH_2), 3.35 (2H, SCH_2), 3.54 (CH_2Cl), 3.91 (OCH, OCH_2), 4.50 (OCH_2O), 4.99, 5.18 (CH_2=), 5.72 (CH=). ^{13}C NMR (75 MHz, $CDCl_3$-d_6, δ ppm): 13.36 (CH_3), 21.92, 29.91, 36.69, 38.46, 45.83 (CH_2), 68.41 (OCH_2), 74.72 (O–CH), 93.73 (OCH_2O), 116.36 (=CH_2), 134.20 (=CH), 223.45 (C=S).



Scheme-V

Synthesis of 3-chloro-2-hydroxypropyl pentyl carbonotrithioate: 1-Bromo-3-chloropropan-2-ol (17.34 g, 0.10 mol) was added to a solution of potassium pentyl trithiocarbonate (26.20 g, 0.12 mol) in acetone (70 mL). The reaction mixture was stirred for 5 h under the reported mild-temperature conditions, then diluted with hexane, washed with water and filtered (**Scheme-VI**). Hexane was removed by distillation and the crude product was purified by column chromatography using benzene/ 2-propanol as the eluent. Yield: 82%; Density (at 20 ºC): 1.2097 g/cm^3; refractive index, (n_D), 1.5916; molar refraction (MR_D, g/mol): calcd. (found): 75.97 (76.14); Elemental analysis of $C_9H_{17}ClOS_3$: calcd. (found) %: C, 39.60 (39.41); H, 6.27 (6.41); S, 35.24 (35.00); Cl, 13.00 (12.81); ^{1}H NMR: (300 MHz, $CDCl_3$-d_6, δ ppm): 0.74 (3H, CH_3), 1.01-1.61 (m, 6H, CH_2), 2.40-3.70 (m, 7H, CH_2S, CH_2Cl, OH), 3.80 (OCH); ^{13}C NMR (75 MHz, $CDCl_3$-d_6, δ



Scheme-VI

ppm): 13.77 (CH_3), 22.13, 27.75, 29.35, 30.65, 37.11, 39.99, 48.33 (CH_2), 69.57 (OCH), 224.12 (C=S).

Tribological activity: The tribological properties of the synthesized compounds were evaluated in MC-20 transmission oil using a four-ball friction tester. The oil is a high-viscosity lubricant formulated from low-sulphur base oils and its technical specifications according to GOST 21743-76 are shown in Table-1. ShKh-15 bearing steel balls, equivalent to AISI 52100 steel, were used for testing. The apparatus consisted of three stationary balls immersed in the lubricant and a fourth ball rotating against them at (1460 ± 70) rpm under the applied load. Each test lasted (10.0 ± 0.2) min. Wear was assessed from the arithmetic mean of the wear-scar diameters measured on the three lower balls. The critical load (P_c) and welding load (P_w) were determined from replicate measurements using the lower recorded values. The scuffing index was evaluated by starting at 196 N (20 kgf) and progressively increasing the load up to the welding load. The scuffing index (I_s), N (kgf), was calculated by the following formula:

$$I_s = \frac{\Sigma Qi}{n} \quad (1)$$

where ΣQi is the sum of the applied loads from the initial load up to the load immediately preceding the welding load and 'n' is the number of tests performed.

RESULTS AND DISCUSSION

1-Bromo-3-chloropropan-2-ol was synthesized by hydrobromination of epichlorohydrin with 40-45% hydrobromic acid at a molar ratio of 1:2 following the method reported by Zeynalov *et al*. [22]. This intermediate was used to prepare butoxymethoxy- and allyloxymethoxy-functionalized derivatives using alcohols, formaldehyde or paraformaldehyde and a KU-2 cation-exchange resin [23,24]. Butyl and pentyl trithiocarbonate salts were obtained by reacting the corresponding thiols with CS_2 and KOH [21]. Subsequent reactions with the brominated intermediates yielded trithiocarbonic acid derivatives containing ether and chlorine functionalities as well as 3-chloro-2-hydroxypropyl pentyl carbonotrithioate [24-26]. The structures of the synthesized compounds were characterized by IR and $^1H/^{13}C$ NMR spectroscopy [27] and their tribological properties were evaluated in MC-20 lubricating oil to assess the influence of functional groups on anti-wear performance.

Effect of functional groups on tribological performance: The tribological performance of the synthesized trithiocarbonate derivatives varied with the nature of the functional groups attached to the molecular structure. The experimental results established the following order of effectiveness: hydroxyl derivative > allyloxymethoxy derivative > butoxymethoxy derivative. Since the compounds share the chlorine containing and sulphur rich trithiocarbonate moiety, this difference points to the role of the additional functional groups in governing surface interactions and protective film formation.

The tribological data (Table-2) establish a clear relationship between the molecular structure of alkyl trithiocarbonate derivatives and their performance in MC-20 oil. At a concentration of 5%, compound **4**, containing a hydroxyl group, exhibited the highest scoring index (I_s = 76) followed by compounds **2** (I_s = 72.8), **3** (I_s = 70) and **1** (I_s = 68)). All four additives performed better than the base oil (I_s = 36)). Compound **1** recorded the highest welding load (4920 N), while compounds **1-4** exhibited the same critical load of 1382 N.

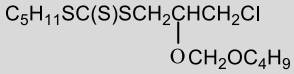
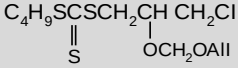
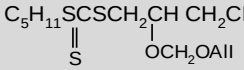
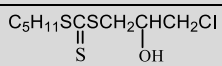
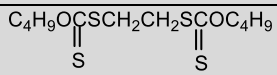
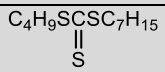
The high scoring index of compound **4** may be associated with its hydroxyl group, which can promote adsorption onto metal oxide/hydroxide surfaces through hydrogen bonding and dipole interactions. Such interactions may strengthen additive retention at the metal-lubricant interface and favour the formation of a protective boundary film. Its critical load of 1382 N and welding load of 3920 N were substantially higher than those of the base oil (784 and 1568 N, respectively). This interpretation agrees with previous studies on hydroxyl-containing lubricant additives [19,28].

Compound **1**, containing a butoxymethoxy group, recorded a scoring index of 68 and the highest welding load (4920 N). Its ether oxygen may facilitate interaction with the metal surface, while the C=C bond of the allyl group may participate in friction-induced chemical reactions. Earlier research [29] reported mechanochemical polymerization of adsorbed allyl groups under frictional conditions, which may contribute to tribofilm formation. Compound **2**, containing a

TABLE-1
TECHNICAL PROPERTIES OF MS-20 OIL

Indicators	GOST standard 21743-76
Kinematic viscosity at 100 ºC, mm^2/s, not less	20.25
Viscosity index, not less	80
Coking %, not less	0.29
Content of selective solvents, water-soluble acids and alkalis, mechanical impurities	Absence
Open cup flash point ºC, not below	265
Freezing point ºC, not above	-18

TABLE-2
RESULTS OF COMPARATIVE TESTS OF ALKYL TRITHIOCARBONATE IN MC-20 OIL

S. No.	Derivatives	Test samples	Concentration of compounds in oil (%)	Tribological properties according to GOST 9490-75			Sulphur content (%)
				Scoring index, I_s	Critical load, P_c, N	Welding load, P_w, N	
1			5	68	1382	4920	1.33
2	Allyloxymethoxy derivative		5	72.8	1382	3724	1.46
3			5	70	1382	3724	1.40
4	Hydroxy derivative		5	76	1382	3920	1.76
5	Butoxymethoxy derivative	 LZ-6/9	5	50	980	309	1.89
6			5	58	1097	3035	1.81
7	–	MC-20	–	36	784	1568	–

allyloxymethoxy group, exhibited a scoring index of 72.8, a critical load of 1382 N, and a welding load of 3724 N. Its ether oxygen atoms may contribute to surface adsorption, while the butyl group may improve oil solubility and influence the orientation of the molecule at the metal interface. Moreover, compound **2** achieved a higher scoring index than compound **1**, although its welding load was lower. The difference in the performance cannot be explained by functional groups polarity or hydrocarbon-chain length alone; as adsorption behaviour and interfacial film formation may also be involved [30].

Compounds **5** and **6** exhibited lower scoring indices of 50 and 58, respectively, with welding loads of 309 and 3035 N. These values suggest that the presence of Sulphur containing functionality alone is insufficient to ensure high tribological effectiveness. Although the sulphur content varied among the tested compounds, but it cannot establish a direct relationship between sulphur content and performance. The results point to the importance of molecular structure and functional-group chemistry in governing the scoring index and load-carrying properties.

Role of trithiocarbonate group and reference additives: The sulphur-rich trithiocarbonate group is another important structural feature shared by the investigated compounds. Under frictional conditions, sulphur containing additives can react with metal surfaces to form protective sulphur containing tribofilms, which potentially reduce the direct metal-to-metal contact, adhesion and wear. The effectiveness of these additives depends not only on sulphur content but also on molecular adsorption, reaction pathways and the composition and durability of the resulting surface film. Ethylene *bis*(butyl xanthate), which contains two xanthate groups and heptyl butyltrithiocarbonate, which has a comparatively simple sulphur containing structure, were used as reference compounds [31].

Effect of chlorine substituent: The chlorine substituent is common to the synthesized derivatives and may contribute to their behaviour under severe frictional conditions. The chlorine containing additives can react with metal surfaces to form chloride containing tribochemical layers, particularly under conditions of elevated contact temperature and pressure [2,32]. Such reactions may influence load-bearing capacity and resistance to scuffing.

Molecular structure and tribological behaviour: The comparative results suggest that the tribological performance of these trithiocarbonate derivatives is governed by the combined effects of the sulphur-rich group, the chlorine substituent and the additional functional groups. Among the compounds examined, the hydroxyl containing derivative showed the better effectiveness followed by the allyloxymethoxy and butoxymethoxy derivatives. The results point to the importance of molecular polarity and surface affinity in tailoring additive performance. Further analysis of the worn surfaces would help establish whether the observed differences arise from changes in adsorption, tribochemical film composition or film durability.

Conclusion

A series of chlorine containing trithiocarbonate derivatives with different molecular structures was synthesized and characterized by IR and NMR spectroscopy. The compounds share a sulphur-rich trithiocarbonate moiety and a chlorine substituent, while differing in the attached hydroxyl (–OH), butoxymethoxy (–$OCH_2OC_4H_9$) and allyloxymethoxy (–$OCH_2OCH_2CH{=}CH_2$) groups. Their evaluation in MC-20 lubricating oil revealed the following order of tribological effectiveness: hydroxyl derivative > allyloxymethoxy derivative > butoxymethoxy derivative. This trend establishes a relationship between molecular structure, functional-group composition and lubrication performance. The superior performance of the hydroxyl-containing compound may be associated with stronger interactions with the metal surface, whereas the ether substituents may influence surface adsorption and compatibility with the oil. The results emphasize that the performance

of trithiocarbonate-based extreme-pressure and anti-wear additives depends on the combined contribution of the sulphur rich core and the attached functional groups.

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CONFLICT OF INTEREST

The authors declare that there is no conflicts of interest regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI. All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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