International Journal of Research in Engineering and Applied Sciences(IJREAS)
Available online at http://euroasiapub.org/journals.php
Vol. 12 Issue 01, January - 2022
ISSN(O): 2249-3905, ISSN(P) : 2349-6525 | Impact Factor: 6.573 |
Thomson Reuters ID: L-5236-2015

Novel Heterocyclic N&S Compounds as E.P. Lubricant Additives

Dr. Akhilesh Kumar Tripathi[1]
Maharishi University of Information & Technology, IIM Road
Lucknow, Uttar Pradesh, India

Prem Shankar Tripathi[2]
B.N. College of Engineering and Technology, Sitapur Road
Lucknow, Uttar Pradesh

Dr. Jitendra Bahadur Singh[3],
N.G. B. University,
Prayagraj, Uttar Pradesh, India

Rajeshwar Rao[4],
Suyash Institute of Information Technology,
Gorakhpur, Uttar Pradesh, India

ABSTRACT

The use of sulphurised natural oils,fats, hydrocarbons, aromatic sulphides and polysulphides, as extreme-pressure and antiwear additives, has already been established. However, the potential of cyclic disulphides merits attention.

The present paper describes the synthesis and evaluation of 0.5% solution of certain cyclic disulphidesas tribological additives, namely 2-benzylideneamino-5-arylimino-3-oxo-1,2,4-thiazolidines in paraffin oil, on a four-ball test rig with 12.7 mm diameter steel bearing balls as test specimens.

All the additives in general, and 2-benzylideneamino-5-(p-ethoxy phenyl)imino-3-oxo-1,2,4-thiadiazolidine in particular exhibited remarkably good antifriction and EP activity, showing a decrease in coefficient of friction and appreciable increase in load-carrying capacity of the lubricant as indicatyed by higher load wear index. The wear surface topography of the used test specimen was studied using scanning electron microscopy.

Keywords: *Antifriction additive, E.P., Lubricant additives*.

INTRODUCTION

Tribochemistry, the chemistry of interacting metallic surfaces, has grown into a well-organized branch of tribology during the last fifty years and has helped in the selection of suitable additive package for a particular use. Additive treated lubricants are extensively used in automotive engines, manual and automatic transmissions and rear axles.

The applications of certain organic compounds and sulphurized vegetable and mineral oils as antiwear and EP lubricant additives have been well recorded (1-4). These additives help to increase efficiency and fuel economy and decrease wear, friction and destructive heating at higher loads.

In view of the above EP additives and their use in industrial, defence and space applications, the reported activity of open-chain disulphides (5-6) and continued efforts in the development of more active and temperature resistant nitrogen and sulphur compounds, this paper reports the synthesis and tribological evaluation of certain 2-benzylideneamino-5-arylimino-3-oxo-1,2,4-thiazolidines as cyclic disulphide system in paraffin oil using 12.7 mm diameter alloy steel balls as test specimen in a four-ball test

EXPERIMENTAL

The Base fluid

The base fluid used was paraffin oil specific gravity 0.81 at 25 ^{0}C, viscosity i) at 40 ^{0}C, 52.30 cP and ii) at 100 ^{0}C, 5.02 cP, cloud point -2 ^{0}C, Pour point -8 ^{0}C, Flash point 185 ^{0}C and fire point 201 ^{0}C.

Additives

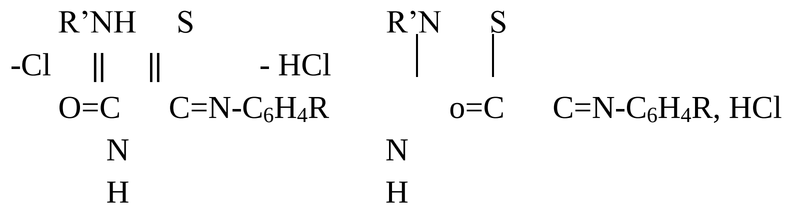
2-benzylideneamino-5-arylimino-3-oxo-1,2,4 –thiadiazolidines were prepared by the interaction of N-chlorosuccinimide with 1-aryl-5-benzylideneamino-2,4-thiobiurets (I) which forms 1-aryl-5-benzylidineaminoi-2-S-chloroiso-2,4-thiobiuret as unstable intermediate (II). The lone pair of electrons on nitrogen atom attacks the sulphur atom of (II)toform another unstable 2-benzylideneamino-5-arylimino-3-oxo-1,2,4-thiadiazolidonium intermediate (III), which simultaneously liberates a proton and affords a stable product, i.e. 2-benzylideneamino-5-arylimino-3-oxo-1,2,4-thiadiazilidine hydrochloride (IV)

o ..
|| R'NH S-Cl↷
HC—C EtOH/Δ | |
R'NH-C-NH-C-NH-C_6H_4R + || N-Cl O=C C=N-C_6H_4R
|| || HC— C N
o o || H
o

(1) (2)

+

International Journal of Research in Engineering and Applied Sciences(IJREAS)
Vol. 6 Issue x, Month - 2016
ISSN(O): 2249-3905, ISSN(P) : 2349-6525 | Impact Factor: 6.573



(3) (4)

Where R= CH_3, Cl, $-OC_2H_5$, $-OCH_3$and R' = C_6H_5-CH=N-

i)2-benzylideneamino-5-(p-methyl phenyl) imino-3-oxo-1,2,4-thiadiazolidine, m.p. 200^0C, $C_{16}H_{14}N_4SO$ **(A_1)**
ii)2-benzylideneamino-5-(p-methoxyphenyl)imino-3-oxo-1,2,4-thiadiazolidine.m.p. 208^0C, $C_{16}H_{14}N_4SO_2$ **(A_2)**
iii)2-benzylideneamino-5-(p-ethoxyphenyl)imino-3-oxo-1,2,4-thiadiazolidine,m.p. 211^0C, $C_{17}H_{16}N_4SO_2$ **(A_3)**
iv)2-benzylideneamino-5-(p-chlorophenyl)imino-3-oxo-1,2,4-thiadiazolidine,m.p. 184^0C, $C_{15}H_{11}N_4SOCl$. **(A_4)**

Reference additive

A standard sulphur containing additive used extensively in EP gear oils was selected as reference additive for comparison. This additive has following physical characteristics:-
Sp. Gravity 0.96 at 25^0C, viscosity 264.95cP at 25^0C, sulphur(wt%) 28.9 min and 32.6 Max.

Specimen

12.7 mm steel balls of the following composition and hardness were used :-
C = 0.25%, Mn = 0.27%, Cr = 0.44, Hardness in VHN = 550

Four ball Test

A variable speed four-ball EP lubricant testing machine operating at 1500 rpm was used to conduct a series of 60 sec. tests (ASTM 2783 & IP 239) until the welding point was reached. The additives (0.5% wt/vol.) were mixed in paraffin oil by stirring and heating at 60 ^{0}C to homogenize the solution.

Table-1

S. No.	Lubricant	(ISL) Kgf	Observation at ISL		(2.5s SDL)	Observation (JBWL)			Weld Load (kgf)	(LWI) kgf	(FTP)
			d mm	µ		Load kgf	d mm	µ			
1.	Plain Paraffin oil Without additive)	85	0.55	0.089	90	176	2.90	0.450	200	26	180
2.	paraffin oil + A1	126	0.75	0.052	140	600	1.59	0.0079	> 600	64	320
3.	paraffin oil + A_2	126	0.70	0.050	140	600	1.54	0.075	> 600	69	389
4.	paraffin oil + A_3	127	0.55	0.045	145	600	1.28	0.070	> 600	73	425
5.	paraffin oil + A_4	126	0.64	0.048	142	600	1.40	0.080	> 600	71	390
6.	paraffin oil + reference additive	112	0.66	0.44	128	225	2.15	0.249	250	60	245

d* = Mean wear-scar diameter, µ* = Coefficient of friction, ISL=Initial Seizer Load, SDL= Seizer Delay Load, JBWL= Just Before Weld Load, LWI= Load Wear Index, FTP= Flash Temperature Parameter.

RESULTS AND DISCUSSION

Mixture of paraffin oil with additives 2-benzylideneamino-5-arylimino-3-oxo-1,2,4-thiadiazolidines and the commercially available reference additive were evaluated as potential multifunctional additives by determining different parameters e.g. wear-scar diameter (WSD), initial sezer load (ISL), weld load (WL), just before weld load (JBWL), coefficient of friction, load wear index (LWI) and flash parameter (FTP). The results were compared with plain paraffin oil and reference additive and are recorded in Table-1.

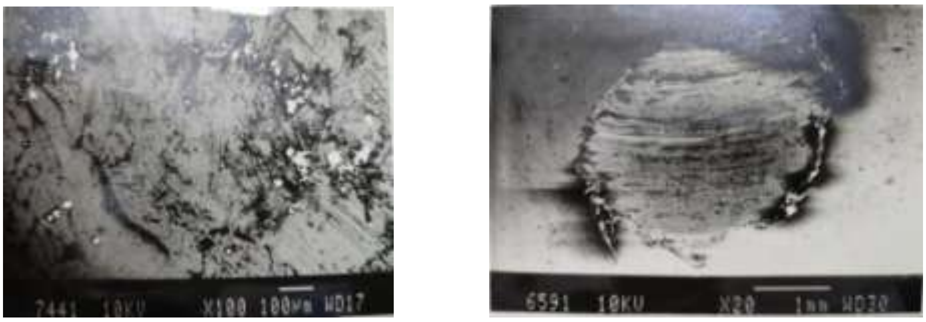
From Table-1, it is clear that all the additives tested were found to be quite effective, as indicated by the increased initial seizer loads and weld loads and marked increase in load wear index and flash temperature parameter. All the additives afford lower wear-scar diameter as compared to the plain paraffin oil and reference additive in wear test. Especially, the additive (A3) i.e.2-benzylideneamino-5-(p-ethoxyphenyl)imino-3-oxo-1,2,4-thiadiazolidine appears to exhibit the best antiwear activity, load carrying characteristics and reduce the coefficient of friction values as compared to plain paraffin oil and even reference additive. The additive A4 also give remarkablyhigh values of LWI, FTP and weld load and offered lower values of wear-scar diameter at JBWL (600 kgf).

Thus, it is obvious that up to the ISL, the load is carried by the physiosorbed thin layer of liubricant additive admixture. Beyond the ISL, the sudden increase in the values of the wear-scar diameter is attributed to the rise in temperature and consequent partial desorption of this physiosorbed layer. Hpwever, at high loads (up to JBWL), a further rise in temperature causes a decomposition of additive and the reactive species formed interact with steel ball leading to the formation of mixed chemisorbed/chemical layer. This thin layer effectively prevents metallic contact between the rotating ball specimen and increase the load carrying capacity.

TRIBOCHEMISTRY

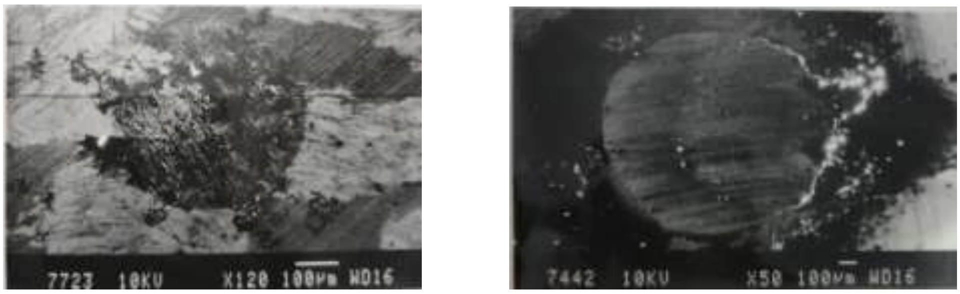
Surface analysis

Topography of the wear-scars produced an steel ball specimen was examined by using Scanning Electron Microscopy (SEM). Figures 1 & 2 depict the SEM micrographs of wear scar for plain paraffin oil and admixture of paraffin oil with 2-benzylideneamino-5-(p-ethoxyphenyl)imino-3-oxo-1,2,4-thiadiazolidine. The flow pattern at the leading edges indicates that the lubricant flowed from left to right. The micrographs appear to indicate adhesive wear. The micrographs at higher loads are comparatively smoother than the micrographs at lower loads. This may be due to the formation of a mixed lubricating film.
This confirms that the additive has better lubricating efficiency at higher loads.



(a) 85 kgf (b) 176 kgf

Figure-1 Scanning Electron Micrograph of Plain paraffin oil



(a) At 127 kgf (b) At 600 kgf

Figure-2 Scanning electron micrographs of 2-benzylideneamino-5-(p-ethoxyphenyl) imino-3-oxo-1,2,4-thiadiazolidine

CONCLUSION

The following conclusions have been drawn:-

i)All the additives prepared were found to exhibit good load carrying capacity.

ii)The additive,2-benzylideneamino-5-(p-ethoxyphenyl) imino-3-oxo-1, 2,4-thiadiazolidine appears to be the best EP Lubricant additive.

ACKNOWLEDGEMENT

Authors are thankful to the Director, Indian Institute of Technology, Banaras Hindu University for providing the facilities required for analysis.

REFERENCE

1.Dowey W. and Edwards E.D., “Wear”, 1(1957/58), p.291

2. Mortier R.M. and Orszulic S.T. (eds), “Chemistry and Technology of Lubricants”, Glascow and VCH, Newyork, (1992)

3. Han X. and Li Q. “Tribol. Trans”., 36 (1993), p283.

4. Forbes E.S., “Tribology”, 3(1970), p 145.

5. Bhattacharya A., Sing T. and Verma V.K., “Tribol. Int.”, 23(5) (1990),p 361.

6. Verma V.K., Singh R.,Bhattacharya A. and Triparhi A,K., “Lubr. Sci”, 13-1,(2000),p37-44

Biography of Authors

	The author is Ph.D in Applied Chemistry from I.I.T. B.H.U / Published 22 research papers in various Journal of repute, and published one book on Engineering Chemistry/ Working in the area of Tribology.
	The author is M.Sc. in Chemistry having one publish paper, working in the area of Tribology.
	The author is Ph.D in electronics Engineering, publish various research papers.
	The author is M.Tech. in Mechanical Engineering. Working as research scholar in the area of Tribology.