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## FULL LENGTH ARTICLE

# Preparation and evaluation of some benzimidazole derivatives as antioxidants for local base oil

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## KEYWORDS

Benzimidazole;  
 Alkyl amines;  
 Base stocks;  
 Oxidation stability;  
 Total acid number

**Abstract** Some benzimidazole derivatives, 2(1-H benzo(d)imidazole-2-yl)thio) N-butyl acetamide Ia, 2(I-H benzo(d)imidazole-2-yl)thio) N-octylacetamid Ib and 2(I-H benzo(d)imidazole-2-yl) thio) N-dodecylacetamide Ic were prepared and studied as antioxidants for base stock. The structure of these compounds was elucidated by elemental analysis, IR and <sup>1</sup>H NMR spectroscopy. The inhibition efficiency of the prepared compounds was determined by studying the oxidation stability of the local base oil via the change in total acid number (TAN), viscosity and infrared (IR) spectroscopy. © 2016 Egyptian Petroleum Research Institute. Production and hosting by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

## 1. Introduction

Motor oil engine is the oil used for lubrication of various internal combustion engines. The main function of motor oil is to lubricate the moving parts; it also cleans, inhibits corrosion, improves sealing and cools the engine parts by carrying heat away from moving parts [1].

The deterioration of lubricating oil often leads to the buildup of insoluble deposits or sludge and consequently the viscosity increases during use. In order to avoid these problems, lubricants need to possess superior oxidative stability. The degradation of lubrication oil resulting in increase in acid-

ity and viscosity reduces the efficiency of the system [2]. Usually lubricating oils operate at higher severities of temperatures and pressure, therefore it's required to improve thermal and oxidative stability and excellent temperature-viscosity characteristics allowing the oil to meet the demanding requirements for use in industrial application [3]. Therefore addition of antioxidants is the suitable way to protect the lubricant from oxidative degradation during industrial application [4].

Sulfur and phosphorous contents of any additive must be used with the lowest level in the formulation of industrial oil [5,6]. Most of heterocyclic compounds which have compact structure possess antioxidant, anticorrosion and antiwear properties [7–9]. Amer et al. [10] studied the effect of some synthesized thiazoles as antioxidant additives for Egyptian lubricating oils. They studied the effect of concentration of the most effective antioxidant in order to obtain the optimum concentration to be used. They concluded that increasing the additive concentration led to a decrease in oxidative products. The

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antioxidant activities of some poly-functionalized phenols linked to heterocyclic derivatives were evaluated [11].

## 2. Experimental

All reagents (purchased from Merk Co., Aldrich and Fluka Chemical Co.) were of analytical grade and used without further purification. The tested base stock was delivered from Alexandria petroleum Company. The physicochemical characteristics of the base stock are tabulated in Table 1.

### 2.1. Preparation of additives (Ia–c)

The additives were prepared [12] according to the pathway outlined in Scheme 1.

#### 2.1.1. Preparation of the ester ethyl-2((1-H benzo(d)imidazole-2-yl) thio acetate)

In a 250-cc round-bottomed flash are placed 15.02 g. (0.1 moles) of 2-mercapto-benzimidazole (I) and 12.25 g of ethyl chloroacetate (0.1 mol). This mixture is then refluxed for three hours in ethanol. The ester is then collected and

recrystallized from n-pentane (with a 70% yield) and its melting point is measured (136–138 °C).

#### 2.1.2. Preparation of additives, 2((1-H benzo(d)imidazol-2-yl) thio) N-alkyl acetamide (Ia–c)

In a 100-cc conical flask are placed 11.8 g. (0.05 mol) of ester and (0.05 mol) of N-alkyl amines, (N-butylamine (a), N-octylamine (b) and N-dodecylamine (c)). The mixture is cooled to zero °C in ethanolic KOH for one hour. The products (Ia–c) were filtered and recrystallized from ethanol. The compounds were characterized by elemental analysis, IR and <sup>1</sup>H-NMR spectrophotometric techniques.

### 2.2. Oxidation stability study

The oxidation test was carried out at 120 °C, according to ASTM D-943 standard method. The oxidation cell in the static mode contained 250 ml. base stock, and activated copper and iron wires catalysts. The base stock sample was subjected to oxidation with pure oxygen (99.95%) at a flow rate of 0.1 L/h for maximum 96 h. The characterized 2-mercaptobenzimidazole and its derivative (Ia–c) compounds were added in different concentrations, ranging from 200 to 1000 ppm. The oil sample after, 24, 48, 72 and 96 h of oxidation times were analyzed for their viscosity, total acid number and infra-red spectroscopy.

#### 2.2.1. Total acid number (TAN), and viscosity

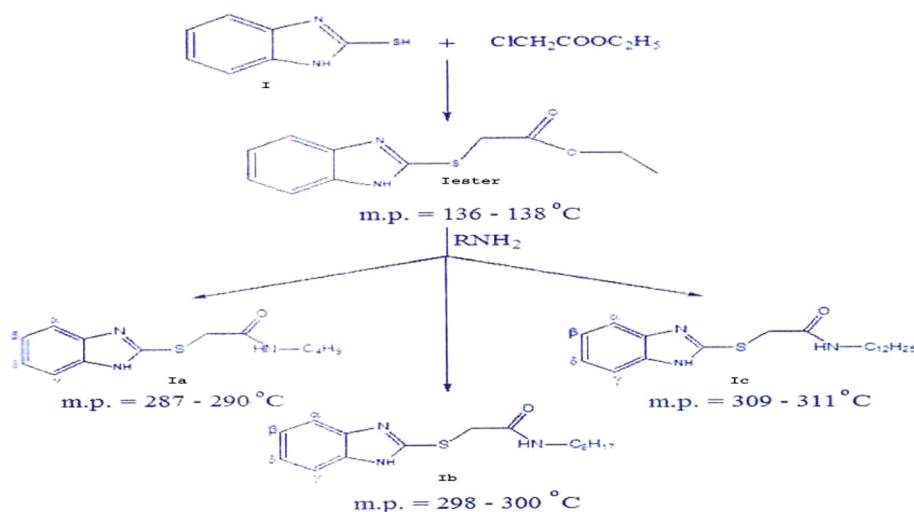
Total acid number and viscosity were carried out according to ASTM standard test methods (D-664 and D-445, respectively).

#### 2.2.2. FT-IR spectroscopy

Infra-red spectra of the oxidized samples at different periods were recorded on FT-IR Spectrophotometer, Model 960 M00g, ATI Mattson Infinity Series, USA. A thin film of the sample, 5 mm thickness and 13 mm diameter and spacers with 0.025 mm thickness (path length) were used. The spectra of the studied samples were measured in the range of 4000–400 cm<sup>-1</sup> with a suitable scan resolution 4 cm and scan rate 32 cm/min. Elemental analyses were carried out in the Micro Analytical

**Table 1** Physicochemical characteristics of base stock.

Test	Test method	Result
Density at 15.5 °C, g/L	ASTMD4052	0.8916
Pour point, °C	ASTMD97	-3
Viscosity 40 °C	ASTMD-445	170.5
100 °C	ASTMD-445	15.03
Viscosity index (VI)	ASTMD-2270	86
Total acid number (TAN)	ASTMD-664	0.025
Sulfur content, wt%	ASTMD-4294	0.53
Carbon residue wt%	ASTMD-524	0.7
Ash content, wt%	ASTMD-482	0.004
Wax content, wt%	UOP 46	0.85
Copper corrosion	ASTMD-190	Ia
Water content, ppm	ASTMD-1744	50



**Scheme 1** Preparation of additives (Ia–c).

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