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Mahmood Ezzulddin Ahmed

University of Mosul

Roua'a Kassim AL-Ojar

University of Mosul

Yonis Ahmed Ismael

University of Mosul

Fawzi Habeeb Jabrail

fawzijabrail@uomosul.edu.iq

University of Mosul

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Synthesis of Lubricant Additives from Polyisobutylene Reclaimed from Inner Tube Waste: Characterization and Application in Automotive Engines

Mahmood Ezzulddin Ahmed¹, Roua'a Kassim AL-Ojar^{2*}, Yonis Ahmed Ismael³, Fawzi Habeeb Jabrail^{4*}

¹Ministry of Education, Directorate of Education in Nineveh, 9523 + P42, Mosul, Iraq.

²Department of Biochemistry, Al-Batool college of Medicine, University of Mosul, Mosul 41002, Iraq.

³College of Pharmacy, Nineveh University, Mosul, Iraq.

⁴Polymer Research Lab, Chemistry Department, College of Science, University of Mosul, Mosul 41002, Iraq.

*Corresponding author.

E-mail addresses: fawzijabrail@uomosul.edu.iq (Fawzi H. Jabrail), rouaa.alojar@uomosul.edu.iq (Roua'a K. AL-Ojar), yonis.ahmed@uoninevah.edu.iq (Yonis A. Ismael), hmooezzu@gmail.com (Mahmood E. Ahmed).

Abstract

Polyisobutylene succinimide lubricating oil additive is manufactured from waste inner tubes, which have shown mainly excellent properties such as oxidation stability, dispersancy, viscosity index, and improved properties like flash point and pour point. The goal of this study is to reclaim polyisobutylene rubber from waste inner tubes by chemical dissolution method using kerosene solvent. After confirming the chemical composition of the polyisobutylene and its crystalline, morphological and thermal properties using FTIR, ¹H NMR, XRD, DSC and FESEM, it was reacted with maleic anhydride to obtain the intermediate compound polyisobutylene succinic anhydride, which in turn was reactivated with diethylene triamine to manufacture the lubricant oil additive polyisobutylene succinimide. The latter was mixed in different proportions with lubricating oil. A number of tests have been performed on the mixture and the 2% mixture has demonstrated a clear improvement in the required qualities of the lubricant.

Keywords: Inner tube waste, polyisobutylene, lubricant oil additives, polyisobutylene succinimide, dispersant, anti-oxidant.

1. Introduction

Accumulation of rubber and plastic waste is considered a global problem, where these polymers represent a large part of solid industrial waste that causes environmental pollution [1]. The lack of proper polymer waste accumulation treatment techniques has led to critical and incurable problems [2, 3]. Synthetic rubbers are hydrocarbons that have limited sources, mostly petroleum; therefore, they must be used wisely rather than wastefully [4]. Accordingly, recycling rubber has become compulsory to save its raw materials and reduce pollution [5, 6]. Recently, different methods have been used for the recycling of rubber and plastic waste and have been designed to be suitable for different polymer composite materials [1, 7].

Butyl rubber (mainly consisting of polyisobutylene with a small amount of polyisoprene) has been used in a wide range of applications, most importantly in tires and others such as tire inner liners, inner tubes, and tire curing bladders due to its low unsaturation [8]. Butyl rubber, more precisely the inner tube of tires, has become obsolete due to its inability to decompose easily and its non-reusability because of its three-dimensional network structure. Therefore, suitable recycling processes need to be applied for the recovery of its materials, including microwaving [9], thermomechanical and mechanochemical devulcanization methods [10], as well as chemical dissolution processes [1, 5]. These methods can be used for recycling butyl rubber, particularly the use of organic processes where the reagents used, such as decalin, xylene, thiophenols, etc., can be applied to the cleavage of sulfur crosslinks (i.e., scission of S – S or C – S bonds) of the vulcanized rubber [11, 12]. The basic constituent substance of butyl rubber can be reclaimed using hydrocarbon solvents, which penetrate among the rubber chains, causing interactions between polymer – polymer and solvent – polymer that encourage the swelling of rubber chains and break them into small segments [5]. Butyl rubber, a copolymer of 98% isobutylene and 2% isoprene, has very good industrial properties such as low gas permeability, chemical and moisture resistance, and good thermal and oxidative stability. Butyl rubber is mainly used in tire applications such as tire inner liners, inner tubes, tire curing bladders, etc. The recovered polyisobutylene has a wide range of applications; it is used in lubricants, adhesives, sealants, etc. Modern lubricants are prepared from a variety of base fluids and chemical additives, where the additives must be included to minimize wear by forming a low shear strength surface film and to prevent rust formation, with concentrations varying from a few ppm to over 30% [13]. The solubility of chemical additives in petroleum products must be maintained over a broad temperature range [14]. The main function of oil additives is indeed to prevent accumulated sludge and deposits in the crankcase within the engine and on the

cylinder walls [15]. Such types of additives are called detergents, which prevent engine damage by cleaning, inhibiting deposit formation, and neutralizing acids [16].

Surfactants and detergents with amphipathic chemical structures at the oil - water interface form micelles in the oil phase and promote the stability of micelles particularly at elevated temperatures, like polyisobutylene succinic anhydride (PIBSA) – based surfactants [17, 18]. The ability of polyisobutylene (PIB) to interact with nonpolar liquids will help the polymer be used in several other applications such as absorbing crude oil; it is also used as a dispersant [19].

The reaction of polyisobutylene (PIB) with maleic anhydride at high temperature via an Alder – Ene reaction will produce polyisobutylene succinic anhydride (PIBSA), which is used in engine oil formulations as the most popular dispersant [20]. Moreover, the reaction of PIBSA with different polyamines in (1:1) or (1:2) ratios forms dispersants called polyisobutylene succinimide (PIBSI) [21]. They are the most well - known non - ionic dispersants, and because of their excellent dispersancy of insoluble oxidation products and low - temperature performance, they are widely used in the oil industry [22].

Using a greater number of secondary amines in the core of the polyamines used in PIBSI dispersants will form a stronger binding dispersant and provede better oil performance [23, 24].

In the present work, polyisobutylene (PIB) was reclaimed from inner tube waste by a chemical dissolution process using a hydrocarbon solvent represented by kerosene one of the petroleum oil derivatives. The recovered PIB was then reacted with maleic anhydride for the preparation of polyisobutylene succinic anhydride (PIBSA) – based dispersants. Finally, oil dispersants were synthesized from the reaction of PIBSA with diethylenetriamine (DETA), resulting in the formation of polyisobutylene succinimide (PIBSI) with a diethylenetriamine bridge. The synthesized lubricating oil dispersants have PIB chains soluble in oil, whereas the succinimide group acts as a polar head. The reclaimed substances and those synthesized from them were diagnosed by various characteristic methods, including FTIR, ^1H NMR, X - ray diffraction, thermal analysis (DSC), FESEM, and their molecular weights ($\overline{MW}$). In addition, the synthesized oil additive blended in different percentages with lubricant oil was evaluated for its kinematic viscosity, antioxidation, and dispensability.

2. Experimental

2.1. Chemicals

Inner tube scrap from a local market was collected and cleaned by washing with water and detergent. The cleaned rubber was dried and ground into particles of about 450 microns in size. Kerosene was supplied by Baiji Refinery, Iraq. The chemicals maleic anhydride (MA) and diethylenetriamine were obtained from BDH Chemical Company (Poole, UK) and were used as received. The solvents methanol and dimethyl sulfoxide (DMSO) were supplied by Fluka

Company (Switzerland) and used without further purification. Silicon dioxide (SiO₂) and aluminum oxide (Al₂O₃) were obtained from Sigma - Aldrich Company.

2.2. PIB Rubber Reclamation

A chemical dissolution method was used for polyisobutylene (PIB) reclamation. A certain weight of inner tube scrap particles, 10.0 g (10.0 wt%), is poured into a 500 ml conical flask. Kerosene, 200 ml, is added, and the flask is closed and fixed inside a water bath that operates at 50 °C for three days. The flask is kept inside the water bath and shaken from time to time. After the determined time, the soaked rubber solution is transferred into a 250 ml round - bottom flask; subsequently, 7.0 g of SiO₂ and 3.0 g of Al₂O₃ are added. The contents of the flask are heated using a mantle with a magnetic stirrer to the boiling point of kerosene (about 200 °C) for 2 hours with stirring. In the end, the heat is stopped, and the solution is left to cool under stirring. The solutions are filtered, and then 30 % (V/V) methanol is added to the filtrate. The mixture is heated at 50 °C inside a water bath and left for sufficient time for rubber precipitation in an oily form, which has a brown color and becomes solid with a dark brown color after two days.

2.3. PIB's Molecular Weight Measurement

The viscosity average molecular weight ($\bar{M}_v$) of the reclaimed PIB is measured, and an Ubbelohde viscometer is used. Different known concentrations (0.01 – 0.03 g/mL) are prepared using DMSO solvent. The intrinsic viscosity (η) of the reclaimed PIB is determined from measuring the intercept of a linear relationship in a plot of reduced viscosity (η_{red}) versus concentration.

The value of the intrinsic viscosity is then applied in the Mark – Houwink equation for the calculation of $\bar{M}_v$

$$[\eta]_{30^\circ\text{C}} = \dot{K} M_v^a \quad (1)$$

Where

$\dot{K} = 0.0158 \text{ cm}^3.\text{g}^{-1}$; and $a = 0.70$ in DMSO solvent at 30 °C.

2.4. Characterization of PIB Rubber

The reclaimed PIB rubber was studied using the following techniques. The FTIR spectroscopy of the main functional groups of PIB with their absorption frequencies was measured using a Bruker Instrument 6300, Germany. The XRD of PIB was measured with a Philips X - ray diffractometer (PW1730) using Cu - K α radiation (40 kV and 30 mA). Moreover, DSC in the thermal analysis was performed using an instrument from Linseis type, chip - DSC - L 66 Ultimate, USA, with a heat rate of 20 °C/ min and a resolution of 0.13. The organic structure of PIB was detected using ¹H NMR by UXNMR, Bruker Analytische Messtechnik GmbH, Germany;

the solvent used was deuterated DMSO. Finally, the FESEM images of the PIB morphological surface were measured using a TESCAN MIRA FESEM, Czech Republic.

2.5. Synthesis of Polyisobutylene Succinic Anhydride (PIBSA)

In a three - necked round bottom flask equipped with a nitrogen gas inlet, 10 g of the reclaimed PIB was added and heating was started using a sand bath. When the solid PIB melted with rising heat, stirring began. When the temperature reached 250 °C, 40 g of maleic anhydride (MA) was added, and the reaction mixture was maintained at 250 °C with continuous stirring for about 10 hours. Nitrogen gas was kept flowing in a slow stream to the reaction mixture during the heating period. At the end, the reaction was stopped, and the mixture was cooled to ambient temperature; the obtained product was honey brown in color.

2.6. Synthesis of Polyisobutylene Succinimide (PIBSI)

In a round - bottom flask with three necks connected to a nitrogen gas inlet, 5.0 g of polyisobutylene succinic anhydride (PIBSA) was dissolved in 50 ml of toluene and kept under continuous stirring. To the mixture, 25 ml of diethylene triamine was added, and stirring and nitrogen gas flow were maintained for 1 hour. The reaction mixture was gradually heated to the boiling point of the solvent, about 120 °C over approximately 2 hours. Finally, to remove the solvent and water from the reaction, the mixture was transferred to an open flask and kept under vigorous stirring conditions. A highly viscous compound with a dark brown - black color was produced.

2.7. Tests to Determine the Validity of PIBSI as an Oil Additive

Different tests were depended on for the evaluation of PIBSI as an oil additive, where in this test three samples were prepared, including lubricating oil samples blended with 1.0 %, 1.5 %, and 2.0 % PIBSI oil additive, called sample 1, sample 2, and sample 3, respectively.

2.7.1. Anti - Oxidant Test

The test includes measuring the oil's ability to neutralize different acids formed during engine operation and the result is called the total base number (TBN). From each sample, 100 ml was subjected to saline water, then to severe oxidation conditions in the presence of strips of iron and copper, and the mixture was kept for 72 hours at 165.5 °C according to the Indiana test method used for oxidation [25].

The TBN of lubricant oil blend represents its capacity to neutralize acids formed during the operation of oil engines due to combustion by - products and oil degradation. TBN is expressed in mg KOH per gram of oil during the oxidation period. Three samples from the examined mixture are taken: one after 24 hours, the second at 48 hours and the final sample after 72 hours.

2.7.2. Kinematic Viscosity

The kinematic viscosity of the lubricant oil blend samples was measured using a capillary tube viscometer. The method utilizes a calibrated capillary viscometer fixed inside a temperature - controlled bath. The kinematic viscosity is measured at 40 °C (KV₄₀), which represents the lubricant's viscosity at a standard temperature of 40 °C. Moreover, it is measured at 100 °C (KV₁₀₀), and the viscosity index (VI) of the tested sample is calculated according to the formula:

$$VI = \frac{(L - KV_{40})}{(L - H)} \times 100 \quad (2)$$

Where KV₄₀ represents the kinematic viscosity of the oil at 40 °C. L represents the kinematic viscosity at 40 °C of reference oil with a VI of zero. H is the kinematic viscosity at 40 °C of reference oil with a VI of 100.

2.7.3. Dispensability Test

The test in lubricant oil analysis represents the ability of the oil to keep contaminants, chiefly soot from combustion, agglomerated and settled down, but only suspended in the oil. The blotter spot test was used to evaluate the ability of the lubricant oil blend to maintain dispersion of contaminants. The method involves putting a few milliliters of the lubricant oil blend, which was used in the antioxidant test, onto filter paper (type Durieux 122), carefully placing a droplet of the oil. The resulting pattern on the filter paper, which has been observed under specific lighting conditions, allows the dispersancy percentage of the oil to be calculated from the following formula:

$$\%Dispersancy = \frac{\text{Diameter of black spot}}{\text{Diameter of the total spot}} \times 100 \quad (3)$$

2.7.4. Density Measurement

The density of lubricant oil blended with additives is measured by determining the mass and volume of the oil sample using the following formula:

$$P = M / V \quad (4)$$

The correlation between density and additive content is that as additive content increases, the density of the lubricant oil increases, but this is not always the case; it depends on the type of additive and the base material.

2.7.5. Pour Point and Flash Point Tests

The pour point of the lubricant oil blended with additives is determined by gradually cooling the oil and observing the temperature at which it ceases to flow. The pour point equipment includes a cooling bath, test jar, and thermometer. The flash point of the lubricant oil blended

with additives is the lowest temperature at which its vapors will ignite when exposed to an ignition source. The Cleveland open cup method is used for measuring the flash point, where the oil sample is heated in an open cup exposed to the air, and the flash occurs when an ignition source is passed over the oil surface.

3. Results and Discussion

Polyisobutylene (PIB) material was reclaimed from inner tube scrap by a chemical dissolution process. Therefore, the reclaimed PIB, before reacting with maleic anhydride (MA), must be characterized for its structure using ^1H NMR and FTIR. The average molecular weight of PIB rubber must be known, along with its crystallinity and surface morphology using X - ray diffraction and FESEM techniques, respectively. Moreover, the thermal stability of reclaimed PIB is important to establish. The reaction of PIB with maleic anhydride and the produced polyisobutylene succinic anhydride (PIBSA) must be characterized by FTIR. Finally, the reaction of PIBSA with diethylene triamine yields lubricant oil additives called polyisobutylene succinimide (PIBSI), which has been characterized by FTIR. Mixing different percentages of PIBSI additives with lubricant oil will produce blended oil, which needs several tests to confirm its dispersibility, antioxidation, kinematic viscosity, and suitability as engine oil, along with its density, pour point and flash point.

3.1. PIB characterization

3.1.1. FTIR spectroscopy

The FTIR spectrum of the reclaimed PIB (**Figure 1a**) shows the absorption frequencies in **Table 1** of some important functional groups. The reclaimed PIB exhibits its $\gamma(\text{C-H})_{\text{str}}$ of the methylene groups >CH_2 at 2921cm^{-1} , 2853cm^{-1} and 1463cm^{-1} . The $\gamma(\text{C-H})_{\text{str}}$ of PIB methyl groups ($-\text{CH}_3$) shows absorption frequencies at 1388 cm^{-1} . The peak at 1714 cm^{-1} , which represents the carbonyl groups $\gamma(\text{C=O})_{\text{str}}$, is possibly due to the oxidation of PIB, or it can be functionalized to incorporate carbonyl groups [26]. The presence of the peak at 1605cm^{-1} indicates the presence of unsaturation.

Table 1 FTIR adsorption frequencies of PIB, PIBSA, and PIBSI.

FT-IR main functional groups									
examined samples	γ (C-H) str methylene-	γ (C-H)str methyl-	γ (C-H)str methine-	γ (C=O)str	γ (C=C)str	γ (C- C)str	γ (C-O)str	γ (N-H)str	γ (C- N)str
wave number ν/cm^{-1}									

	2921					1230			
PIB	2853	1388	----	1714	1604	1162	1008	-----	-----
	1463								
	3058								
PIBSA	2916	1457	1431	1704	1633	1218	966	-----	-----
	2874								
PIBSI	2936	1388	1308	1617	1561	1239	1038	3382	1345
						1185			1119

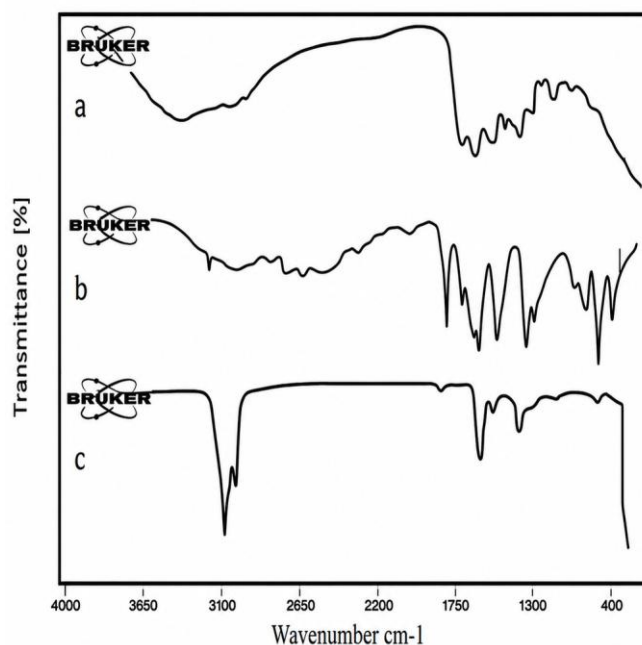


Figure 1: FTIR spectrum of: a-reclaimed PIB, b- synthetic PIBSA, and c- synthetic PIBSI.

$\gamma(\text{C}=\text{C})_{\text{str}}$ groups, which is due to the existence of some degree of unsaturation along the polymer backbone or each PIB polymer precursor moiety having at least two unsaturated end groups $-\text{CH}_2-\text{CH}(\text{CH}_3)_2=\text{CH}_2$, $\text{CH}_2=\text{C}(\text{CH}_3)_2$ [27].

3.1.2. ^1H NMR Spectroscopy

The ^1H NMR spectrum (**Figure 2**) of the reclaimed PIB illustrates the chemical shifts of the PIB protons, which are detailed in Table 2. The proton resonance (singlet, H) at chemical shift

$\delta=1.23\text{-}1.53$ ppm is attributed to the methylene group >CH_2 in the PIB polymer backbone. Additionally, the proton resonance (singlet, H) at $\delta =0.91\text{-}1.17$ ppm corresponds to the methyl groups ($-\text{CH}_3$). The chemical shifts $\delta =3.48\text{-}6.28$ ppm appear as weak signals indicating the presence of vinylic protons ($\text{C}=\text{C}-\text{H}$) and protons attached to oxygen ($\text{CH}-\text{O}$) formed during the recycling of PIB [28].

Table 2: ^1H NMR chemical shifts and proton description of reclaimed PIB from inner tube.

Sample	Chemical shift δ / ppm	Description of protons
Reclaimed	1.23 – 1.53	Methylene (>CH_2) group
PIB from	0.91 – 1.17	Methyl ($-\text{CH}_3$) group
Inner tube	3.48 – 6.98	($\text{C} = \text{C} - \text{H}$) and ($\text{CH} - \text{O}$)

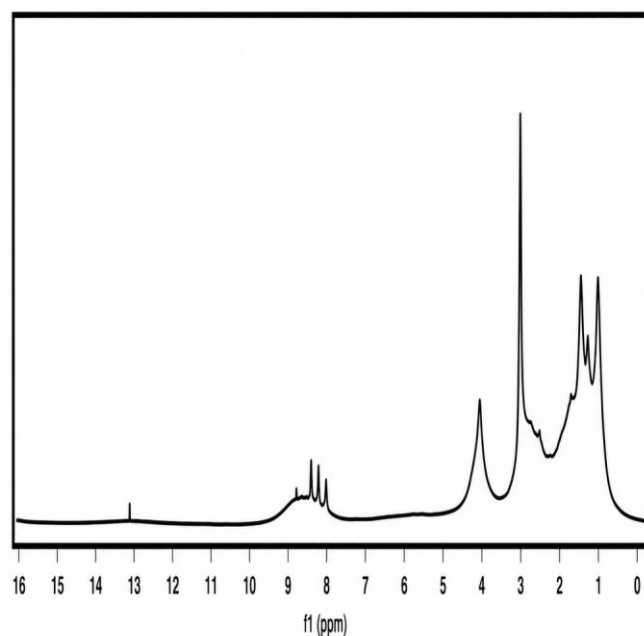


Figure 2: ^1H NMR Spectrum of reclaimed PIB from inner tube.

3.1.3. DSC analysis

The differential scanning calorimetry (DSC) of the reclaimed PIB (**Figure 3**) shows an endothermic peak, starting at 146.3 °C and offset at 159.9 °C, with a peak maximum at 155.0 °C. The area under the peak (**Figure 3**) represents the enthalpy change $\Delta H_f = -0.1 \mu\text{Vs/mg}$ of the transition. The endothermic peak corresponds to the heat absorbed during the transition [29].

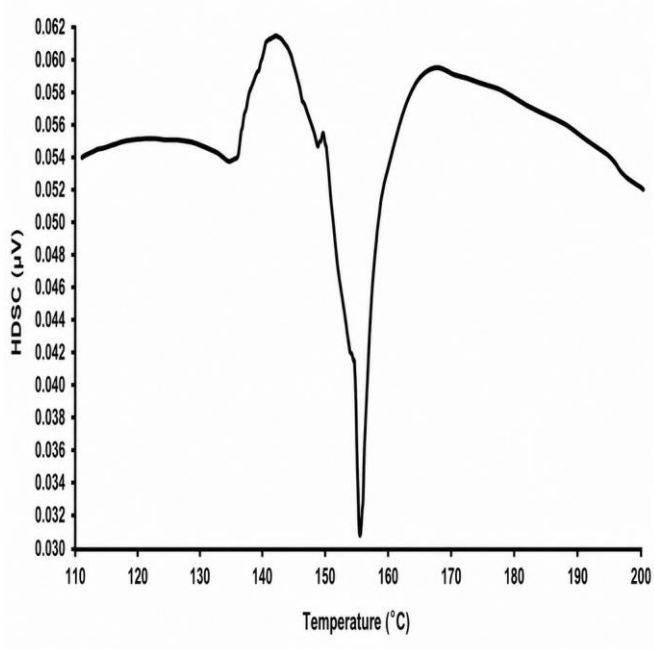


Figure 3: DSC thermogram of the reclaimed PIB from inner tube.

3.1.4. X –ray diffraction

The XRD pattern (**Figure 4**) confirms that reclaimed PIB from the inner tube has a broad peak suggesting amorphous or disordered regions. The XRD pattern also shows small crystalline peaks, likely due to the recycling process. The presence of different small crystalline peaks indicates a low degree of crystallinity, which is attributed to impurities or defects that occurred during recycling [30].

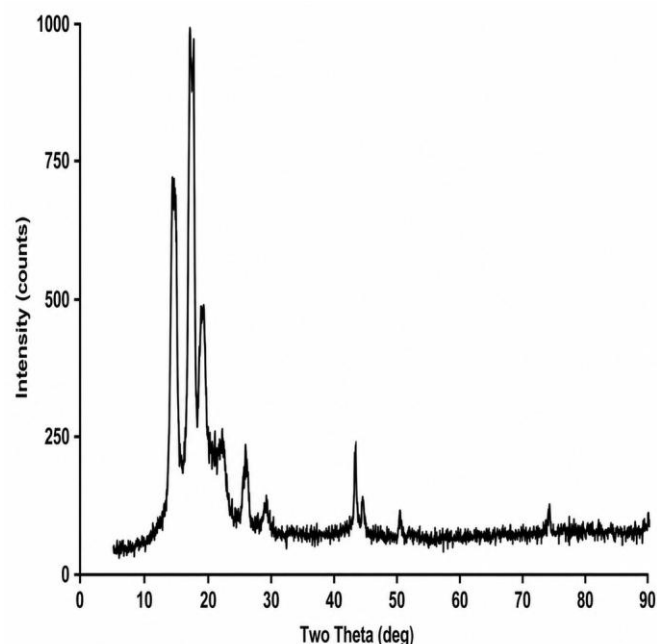


Figure 4: XRD pattern of the PIB reclaimed from inner tube.

3.1.5. FESEM test

The FESEM images of the reclaimed PIB (**Figure 5(a and b)**) show the surface morphology and material microstructure. Studying the surface morphology and texture of the PIB, the FESEM image (**Figure 5a**) with 100 Kx magnification reveals a heterogeneous surface morphology with no uniform particle shapes. The image shows a rough surface texture that denotes that the recycling process of PIB from the inner tube causes changes in the microstructure of PIB. These changes, which are defects in the microstructure of the reclaimed PIB, indicate incomplete recovery of the rubber chains during the recycling process, which leads to reduced mechanical strength compared to the fundamental PIB.

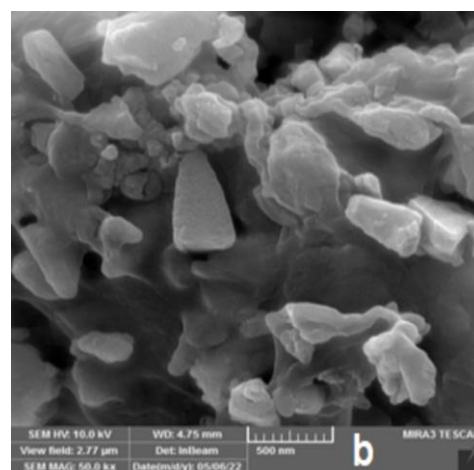
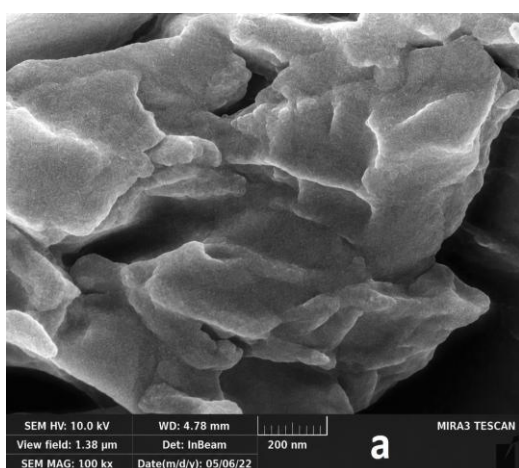


Figure 5: FESEM images of PIB reclaimed from inner tube, a- 100kx magnification; b- 50kx magnification.

The FESEM image (**Figure 5b**) with 50 Kx points out a broad particle size distribution aggregating alongside smaller, finer particles that have a different degree of luminosity. The white spots represent the crystalline particles, which are attributed to impurities or defects from the recycling process [31].

3.2. Molecular Weight determination

The average molecular weight (M_v) of the reclaimed PIB was determined, and the Mark-Houwink equation (Eq. 1) was used for calculation. The intrinsic viscosity was measured from the Mark-Houwink plot, and it was 47 mL/g

$$[\eta]_{30^\circ\text{C}} = \dot{K} M_v^a$$

$$47 = 0.0158 M_v^{0.70}$$

$$M_v = 135,633 \frac{\text{g}}{\text{mol}} \text{ or } (Da)$$

$$M_v = 135.6 \text{ KDa}$$

It seems the molecular weight (MW) of the reclaimed PIB is high, while low MW PIB is favored, especially those that have a high percentage of exo-olefin groups because they are more reactive with maleic anhydride in the Alder-ene reaction, the first step in the production of PIBSA. The efficiency of the reaction of PIB with maleic anhydride becomes less as the MW of PIB increases [32].

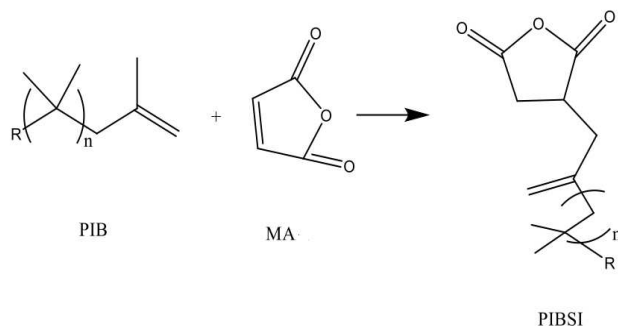
3.3. FTIR analysis and tests examination of the synthesized additives for hydraulic oil

3.3.1. FTIR analysis of PIBSA and PIBSI

Polyisobutylene (PIB), the reclaimed polymer, was reacted with maleic anhydride (MA) in an Alder-ene reaction. The cycloaddition reaction occurs between the alkene such as maleic anhydride (MA), and a compound with allylic hydrogen, such as PIB. The addition of the maleic anhydride group to the polyisobutylene chain forms polyisobutylene succinic anhydride (PIBSA) (**Scheme 1**) [33].

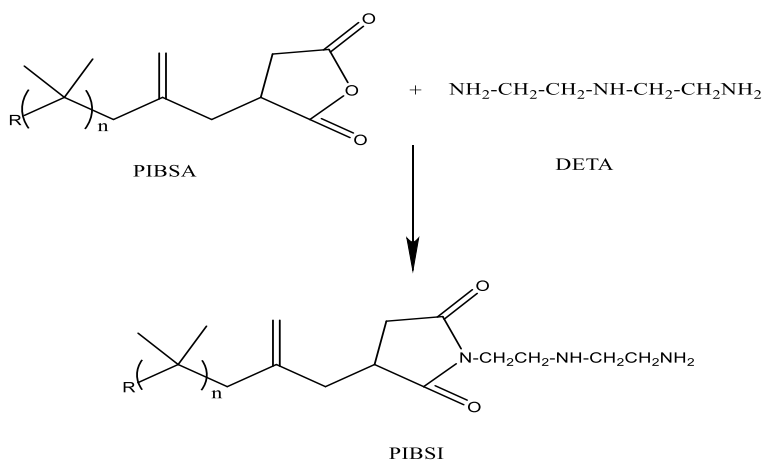
The FTIR spectrum (**Figure 1b**) points out the main peaks of PIBSA with their absorption frequencies (**Table 1**). The peaks at 3058 cm^{-1} , 2916 cm^{-1} , and 2874 cm^{-1} belong to $\gamma(\text{C-H})_{\text{str}}$ of methylene groups present in PIBSA. The absorption bands at 1457 cm^{-1} and 1431 cm^{-1} represent $\gamma(\text{C-H})_{\text{str}}$ of methyl and methine groups, respectively, present in PIBSA. The peak at 1704 cm^{-1} corresponds to $\gamma(\text{C=O})_{\text{str}}$ of carbon groups in PIB. The absorption band at 1633 cm^{-1} belongs to $\gamma(\text{C=C})_{\text{str}}$ of the olefinic double bonds, which are located at the chain ends and are of vinylidene

types [34]. The absorption bands at 1218 cm^{-1} and 966 cm^{-1} belong to $\gamma(\text{C-C})_{\text{str}}$ and $\gamma(\text{C-O})_{\text{str}}$ present in PIBSA.



Scheme 1: Synthesis of PIBSA.

The intermediate compound polyisobutylene succinic anhydride (PIPSA) was reacted with diethylene triamine (DETA) to form polyisobutylene succinimide (PIBSI) (**Scheme 2**), through an imidization reaction [35]. The presence of the PIBSI product serves as a dispersant in lubricants and fuels, where the PIB chain provides oil solubility, while the imide group is used to disperse contaminants and can prevent them from precipitating on engine parts [36,37].



Scheme 2: Synthesis of PIBSI.

The FTIR spectrum (**Figure 1c**) shows the absorption bands of PIBSI with their frequencies (**Table 1**). The peaks at 2936 cm^{-1} and 2872 cm^{-1} represent $\gamma(\text{C-H})_{\text{str}}$ of methylene groups, whereas the absorption bands at 1388 cm^{-1} and 1308 cm^{-1} are for $\gamma(\text{C-H})_{\text{str}}$ of methyl and methine groups. In addition, the peak at 1617 cm^{-1} is for $\gamma(\text{C=O})_{\text{str}}$ of carbonyl groups. The peak at 1561 cm^{-1} belongs to $\gamma(\text{C=C})_{\text{str}}$ of the olefinic bonds, while the peaks at 1239 cm^{-1} and 1185 cm^{-1} are of

$\gamma(\text{C-C})_{\text{str}}$ along the polymer chain. The absorption bands at 1038 cm^{-1} and 3342 cm^{-1} refer to $\gamma(\text{C-O})_{\text{str}}$ and $\gamma(\text{N-H})_{\text{str}}$ of PIBSI. Moreover, the peaks at 1345 cm^{-1} and 1119 cm^{-1} belong to $\gamma(\text{C-N})_{\text{str}}$ of PIBSI oil additive material.

3.3.2. Antioxidant test of PIBSI/ lubricant oil mixture

The antioxidant properties of polyisobutylene succinimide (PIBSI) mixed with lubricant oil are measured according to the total base number (TBN), which indicates the alkalinity of PIBSI and its ability to neutralize acidic byproducts that appear during engine operation. In this test, three samples were mixed with 1.0%, 1.5%, and 2.0% PIBSI in lubricant oil. According to the Indiana test method used for oxidation of the samples, the following results were recorded in **Table 3**.

Table 3: The total base number (TBN) is obtained from titration of the consumed samples with KOH base, expressed in mg KOH per gram of oil at different oxidation times.

Time (h)	TBN in mg KOH/ g of oil			
	Control sample	Sample 1	Sample 2	Sample 3
	0.0%	1.0%	1.5%	2.0%
0.0	10.0	10.0	10.0	10.0
24.0	4.0	7.5	8.0	9.0
48.0	1.0	5.0	6.0	8.0
72.0	0.4	1.2	3.5	5.0

The data in **Table 3** indicates that the TBN values are highest at zero time and gradually decrease over time. Generally, the control sample (**Figure 6** and **Table 3**) show the fastest TBN drop, reaching 0.4 mg KOH/g after 72 hours. This indicates poor acid neutralization and quick depletion of alkaline reserves. The control sample without additives appears to lose its ability (**Figure 6**) to neutralize acids quickly. Sample 1 shows some improvements over the control sample, ending at 1.2 mg KOH/g (**Table 3** and **Figure 6**). Still, the TBN drops relatively fast, showing limited oil additive performance. On the other hand, Sample 2 performs better,

stabilizing at 3.5 mg KOH/g after 72 hours, (**Table 3**, and **Figure 6**), indicating moderate resistance to acid buildup.

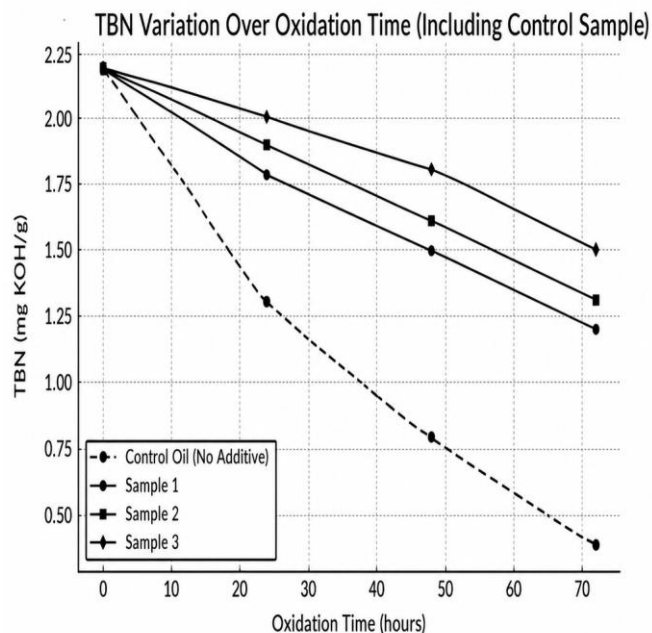


Figure 6: TBN Variation values over oxidation time.

The highest TBN retention was marked for Sample 3, where it reaches 5.0 mg KOH/g. Therefore, Sample 3 records the best acid-neutralizing ability, and its mixture possibly contains a suitable additive percentage.

3.3.3. Kinematic viscosity test of PIBSI/ lubricant oil mixture

In general, viscosity is a measure of a liquid's internal resistance to flow. Viscosity, like many properties, can be described.

3.3.3.1. Viscosity stability and high-temperature performance

Viscosity stability is defined as the ability of a fluid or liquid to maintain its viscosity or resistance to flow over time or under different conditions. Viscosity stability plays a decisive role in determining the effectiveness of lubricant oil under different temperature conditions. In this research, viscosity stability was measured at 40°C and 100°C according to ASTM D445, which is used in kinematic viscosity **Table 4** evaluates the thermal stability of the PIBSI/lubricant oil mixture.

Table 4: Physical properties of PIBSA/ lubricant samples with demonstration of their performance.

Sample	Viscosity stability at		Viscosity index (VI)	Density Kg/m^3	Pour point (°C)	Flash Point (°C)
	40°C	100 °C				
	(cSt)	(cSt)				
1	40.9	6.3	101	819	-9	216
2	44.5	7.8	108	848	-16	219
3	58.6	9.5	115	894	-21	223

The kinematic viscosity of the PIBSI/lubricant oil mixture indicates that Sample 3 shows the highest viscosity at 40°C and 100°C. Accordingly, Sample 3 maintains its film thickness more effectively at elevated temperatures, thereby providing superior protection against wear and thermal degradation [38]. Conversely, Sample 1 shows the lowest viscosity at 100°C, denoting a greater susceptibility to thinning, which means compromising the efficiency of lubrication in high-temperature applications.

3.3.3.2. Viscosity index (VI)

Viscosity index (VI) is a dimensionless parameter that indicates the change in viscosity with temperature. Higher VI values indicate less susceptibility to temperature changes, and vice versa for those with a lower VI.

According to ASTM D2270, the VI was calculated using Eq. 2, and the results showed (**Table 4**) that the highest VI was Sample 3 which indicates that the mixture has better viscosity retention across a wide temperature range. In other words, Sample 3 is the most thermally stable mixture. Therefore, it is suggested that Sample 3 is perfectly suitable for applications involving fluctuating temperature conditions, where the stability of viscosity is critical to maintaining optimal lubrication [39].

3.3.4. Dispensability test of PIBSI/ lubricant oil mixture

The spot dispersancy test (SDT), also known as the blotter spot test, is a simple method used to evaluate in-service engine oil's ability to suspend contaminants like soot.

The test depends on a drop of oil placed on filter paper; the filter paper absorbs all components of the oil and spreads out. The filter paper is placed horizontally on a level surface, like an empty container, and allowed to dry completely after 24 to 48 hours. For characterization of the

prepared dried oil spot on the paper, fresh oil or standard examples are used for comparison. According to the following observations, the dried oil spot can be analyzed [19]:

- a. A fresh, clear, and light spot denotes good oil.
- b. A uniformly dark stain may indicate that the oil is not new, but it can still be used; however, it needs immediate change if the spot is very dark.
- c. A spot of visible particles or debris means the oil is contaminated.
- d. A spot that shows a light periphery around the dark center of the stain means the oil is contaminated with water.
- e. A black spot in the center indicates dirt contamination.

Table 5: Dispersancy of lubricant oil sample and its blend containing PIBSI additive after different oxidation periods.

Sample	Dispersancy at different oxidation periods (mm)		
	24h	48h	72h
Control (unused oil)	34	32	30
Used oil from the rest of the antioxidant test			
1	61	65	69
2	64	68	72
3	70	74	78

The lubricant oil alone, without additives (**Table 5**), shows high dispersancy using Eq. 3 at the beginning of oxidation (24h) when it is fresh; then it decreases after 48h and decreases further at 72h because of oxidation and the formation of solid particles. On the other hand, the used oil obtained from the antioxidant test blended with different percentages of PIBSI lubricant oil additive shows excellent dispersancy power after different oxidation periods (**Table 5**). The results denote that the addition of PIBSI not only disperses the oil contaminants in the oil and thus prevents their accumulation and deposition on the metal parts of the engine, which can

cause damage, but also slightly neutralizes the acidic products resulting from oxidation due to their basic nature [25, 40].

The efficiencies of lubricant oil blended with PIBSI as a dispersant become clear, and its values increase as the percentage of PIBSI increases in the lubricant oil and as the oxidation time period is extended (**Table 5**) [25, 19].

3.3.5. Density test of PIBSI/ lubricant oil mixture

Density serves as the main indicator of the composition of the lubricant, especially with regard to the concentration of the added material to enhance performance. The density of the mixture for the three samples was measured using Eq.4 in accordance with ASTM D4052. Accordingly, Sample 3 in **Table 4** exhibited the highest value, suggesting an increased presence of PIBSI additive components, potentially including dispersants, kinematic viscosity, and antioxidants that contribute to the superior performance characteristics of the blend mixture. At the same time, the lower density of Sample 1 refers to a relatively low concentration of PIBSI additive, which may explain its lower resistance to oxidation, reduced viscosity and depressed dispersancy [19, 41].

3.3.6. Pour Point and Flash Point Tests

Additives in lubricant oil can significantly enhance pour point and flash point. Adding polymers to lubricant oil can depress the pour point at which the oil flows by preventing the formation of wax crystals; at the same time, these polymer additives can increase the flash point by modifying the oil's volatility, where its vapors ignite. The type of additives and their concentrations determine the size of these property changes [42, 43]. Sample 3 shows the highest flash point and lowest pour point (**Table 4**) because PIBSI has a high molecular weight which can reduce the volatility of the lubricant oil and effectively remove more volatile components, lowering the boiling point and thus raising its flash point [19].

At the same time, PIBSI depresses the pour point by interfering with the wax crystals present in the lubricant oil at low temperatures, where the hydrophobic PIB chains can interact with wax crystals and, as a result, prevent the formation of larger solid structures that impede oil flow [44].

4. Conclusions

Polyisobutylene (PIB) was reclaimed from the inner tubes consumed using a chemical dissolution method. A cheap solvent, kerosene, was used in the process of rubber extraction, which was separated from carbon black and the rest of the materials using aluminum oxide and silicon oxide adsorbent materials. Polyisobutylene is obtained in a solid state and has a dark brown color in a ratio of 20 parts per hundred parts of rubber, and a molecular weight of 135.6

kDa. The reclaimed rubber PIB was reacted with maleic anhydride (MA) for the preparation of an intermediate compound, polyisobutylene succinic anhydride (PIBSA), of honey brown color. The latter was reacted with diethylene triamine for the synthesis of a lubricant oil additive called polyisobutylene succinimide (PIBSI).

The synthesized PIBSI was mixed in different percentages with lubricant oil and characterized chemically by applying an antioxidant test. Moreover, the lubricant oil additive was characterized physically using viscosity stability, viscosity index (VI), dispersancy, density, pour point, and flash point tests.

An additive sample of 2% PIBSI added to lubricant oil was shown to be a suitable mixture, having excellent dispersing power for contaminant particles and demonstrating high viscosity, viscosity index, and density, along with the best ability for acid neutralization of solid particles that appeared during engine operation. In addition, the 2% PIBSI can minimize the pour point and elevate the flash point of the lubricant oil.

CRedit authorship Contribution statement

F. H. Jabrail: Conceptualization; Methodology. **R. K. Al-Ojar; M. E. Mahmood; Y. A. Ismael:** Investigation; Validation; Data Curation. **F. H. Jabrail:** Writing - Original draft preparation; Visualization.

Data availability statement

Original data are available on request.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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