Synthesis and Characterization of Novel Terpolymers as Viscosity Index Improvers Using Industrial Waste for Iraqi Lubricating Oil

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Abstract: *The potential utilization of industrial waste, specifically polyethylene wax, to enhance the efficiency of alkyl acrylate polymers as viscosity index improver (VII) additives in lubricating compositions has been investigated. Four terpolymers (w1, w2, w3, and w4) were synthesized via free radical polymerization, incorporating hexyl acrylate, 1-tetradecene, and industrial waste-sourced polyethylene wax at varying molar ratios. These terpolymers were characterized through gel permeation chromatography (GPC) for molecular weight determination, Fourier-transform infrared spectroscopy (FTIR), proton nuclear magnetic resonance (¹H-NMR) spectroscopy for chemical structure analysis, and thermal gravimetric analysis (TGA) for thermal stability assessment. The study evaluated the efficiency of these terpolymers as VII in combination with free additives Iraqi lubricating oil (base oil 60). Our results demonstrate the feasibility of significantly enhancing the efficiency of viscosity improvers through the utilization of industrial waste, with improvements proportional to terpolymer concentration. The terpolymer w3, characterized by a balanced molar ratio of 1:1:1, exhibited the highest degree of efficacy, which gave VI of 206 at 5 wt.% concentration compared with free additive lubricating oil, which gives a VI of 98. This study offers promising insights into the sustainable use of industrial waste to improve lubricating compositions.*

Keywords: *kinematic viscosity; lubricating oil; polyethylene wax; terpolymer; viscosity index improvers*

■ INTRODUCTION

Lubricating oils include a wide range of substances known for their crucial role in reducing friction and ensuring the efficient operation of machinery and engines. These oils consist of various components derived primarily from the residual, highly viscous fraction of crude oil after the distillation process [1-2]. To meet the rigorous standards of original equipment manufacturers, modern lubricants undergo meticulous engineering involving the incorporation of various crucial liquids and chemical additives [3-7]. Most commercially available lubricants are constructed as formulated oils, consisting of base stocks alongside performance-enhancing additives. When these constituents are appropriately present in their specified proportions, they bestow upon the formulated lubricant the requisite properties essential for effective performance within the designated application. Beyond their primary roles encompassing lubrication, cooling, containment/suspension, corrosion prevention, and power transmission, these lubricants are further obligated to fulfill specific functions that are distinctive to the particular application [3].

Several additives can be utilized in diverse applications to raise the performance of lubricants. These additives encompass corrosion inhibitors, viscosity index improvers (VII), antioxidants, pour point depressants (PPD), dispersants, and detergents [7-11]. The lubricant's viscosity is one of the most essential factors to consider when choosing a lubricant. If the lube oil's viscosity is very low at the working temperature, the lube

oil film cannot form correctly, resulting in wear, burning, and sticking. If it is very high, its viscosity resistance rises, causing temperature friction to rise and, as a result, unusual power loss [6]. Because of that, VII and PPD are important additives in lubricant oil.

This study focuses on VII. Oil with elevated viscosity is more viscous compared to that with lower viscosity. When subjected to higher temperatures, oil tends to become less viscous and flow more easily, and conversely, at lower temperatures, it becomes more viscous [6,12]. The alteration in viscosity due to temperature fluctuations is quantified using a parameter known as the viscosity index (VI). A higher VI value indicates that the lube oil's viscosity remains relatively stable across various temperature variations [1]. VII, referred to as viscosity modifiers (VMs), are introduced into lubricating oils to enhance the VI [5,12].

Various polymeric materials have been proposed as potential additives for lubricating oils [3-4,13-14]. VII are characterized by their long hydrocarbon side chains and high molecular weight [15-18]. The functionality of these additives is predicated on their ability to increase an oil's relative viscosity at high temperatures as opposed to low temperatures. Typically, this outcome arises from an alteration in the physical arrangement of the polymer as the temperature of the mixture increases. The polymer molecule in the solution is present in the form of a disordered coil [19], which is swollen by the lube oil solvent. The solubility of polymers typically exhibits an upward trend with the rise in temperature due to the transformation of the polymer molecule from compact coils to a more expanded configuration, resulting in a larger overall volume. This higher volume increases lubricating oil viscosity, which offsets the normal decrease in viscosity with rising temperature [20-21]. The volume of polymer in an oil solution increases as the molecular weight of the polymer increases [22]. Thus, compared to a lower molecular weight polymer of the same kind, a higher molecular weight polymer will impart a greater VI [2].

Methacrylate, acrylate, olefin, and styrene-butadiene polymers and terpolymers are some of the earliest VII [3]. The degree of VI improvement from these polymers is a function of the molecular weight distribution, solubility, and shear resistance [9,23-24]. Many researchers have prepared and studied the high molecular weight polymer viscosity improvers using various monomers, including biodegradable materials, waste products, natural products such as sunflower oil, and manufactured ones such as styrene and maleic anhydride [23-33].

Polymethacrylate was the first viscosity improver that has been studied and developed. The original work was conducted under the supervision of Herman Bruson, who was employed by the Rohm and Haas Company [34]. Afterward, many researchers developed the efficiency of these additives in different methods [9,35-36]. This study focuses on using polyethylene wax, an industrial waste product, to enhance the efficiency of polymethacrylate viscosity improvers. Polyethylene wax, with its high molecular weight and extended hydrocarbon chains, offers a cost-effective means to improve polyacrylates' VI vital components in modern lubricants. This study explores various molar ratios of polyethylene wax and hexyl acrylate to determine the optimal ratio for maximum efficiency when combined with lubricating oil. Additionally, we investigated the impact of varying concentrations of this additive on oil performance.

■ EXPERIMENTAL SECTION

Materials

The following chemicals were used, i.e., hexyl acrylate ($C_9H_{16}O_2$, ≥ 95% purity Merck, Germany), benzyl peroxide ($C_{14}H_{10}O_4$, 25% H_2O purity Merck, Germany), 1-tetradecene (C_7H_8, 97% purity Merck, Germany), and toluene ($C_{14}H_{28}$, ≥ 99% purity Merck, Germany). The polyethylene wax was supplied by The General Company for Petrochemical Industries in Basrah/Iraq. The free additives medium viscosity lube oil (Base oil 60) from the Al-Daura Refinery/Iraq was used in this study. Table 1 shows the physicochemical properties of lubricating oil.

Instrumentation

Spectroscopic analysis for the purified terpolymers was performed in this work. The Fourier-transform infrared (FTIR) spectrometer (FTIR Affinity-1, Shimadzu, Japan), was used to record FTIR spectra of all

Table 1. The physicochemical characteristics of lube oil

Specification	Base oil 60	ASTM methods
Kinematic viscosity at 40 °C (cSt)	66.5	D-445
Kinematic viscosity at 100 °C (cSt)	8.84	D-445
VI	105	D-2270
Pour point (°C)	−6	D-97

terpolymers in the region between 400–4000 cm^{-1} by using a KBr disk. FTIR measurement was conducted to identify the most important functional groups in the terpolymers chemical structure. The structure of the prepared terpolymers was investigated also using proton nuclear magnetic resonance (^{1}H-NMR) spectra obtained with a BRUKER 400 MHz (Germany) equipment using deuterated chloroform (CDCl$_3$) and deuterated dimethyl sulfoxide (DMSO-d_6) as solvents. Thermal analysis (TGA) experiments were conducted using TGA Q500 V6.7 (Germany) at a 10 °C min^{-1} heating rate for a temperature range of 25 to 800 °C under a nitrogen atmosphere with a flow rate of 25 mL min^{-1}. Gel permeation chromatography (GPC) was used to determine the molecular weights of variously produced terpolymers. The analysis was conducted at 25 °C with a 1 mL min^{-1} flow rate and a sample volume of 50 L on a column type 300 mm Jordy Gel DVB mixed. Cannon-Fenske Routine Viscometer was used for fast and easy viscosity testing of the materials.

Procedure

Synthesis of terpolymer additives

The terpolymers were synthesized by free radical polymerization of hexyl acrylates with 1-tetradecene and polyethylene wax with various molar ratios, as shown in Table 2. The mixture and 1% w/w of initiator benzoyl peroxide was dissolved in 25 mL of anhydrous toluene. The mixture was charged into a 250 mL three-neck round bottom flask equipped with a condenser, mechanical stirrer, and glass tube as an inlet section. For 15 min, the reaction mixture was flushed with argon gas. The argon flow rate was then regulated and progressively heated to reflux temperature. The reaction was controlled for 8 h [1,37]. The reaction was then cooled to room temperature, and the terpolymer was purified by methanol washing, filtering, and washing with 20 mL of dried acetone, followed by vacuum drying at room temperature for 24 h. Fig. 1 shows the terpolymer chemical structures prepared from hexyl acrylate, 1-tetradecene, and polyethylene wax. The terpolymers were coded as w1, w2, w3, and w4, which referred to the same terpolymer (hexyl acrylate – 1-tetradecene – polyethylene wax) with various molar ratios of polyethylene wax.

Solubility determination

The solubility of the terpolymers was tested by dissolving them in a free-additive's lubricating oil (base oil 60). In a suitable-sized conical flask, 1 g of the terpolymer was mixed with 100 g of base oil. The resulting combination was allowed to stand for one night. This amount of time was sufficient to cause the polymer to expand. The conical flask was immersed in an oil bath on a thermostated magnetic stirrer hot plate. The oil bath temperature was then raised to 60 °C.

ASTM characterizations

Many standard test methods ASTM were used to determine and evaluate the lubricating oil properties

Table 2. The terpolymer's composition and yield

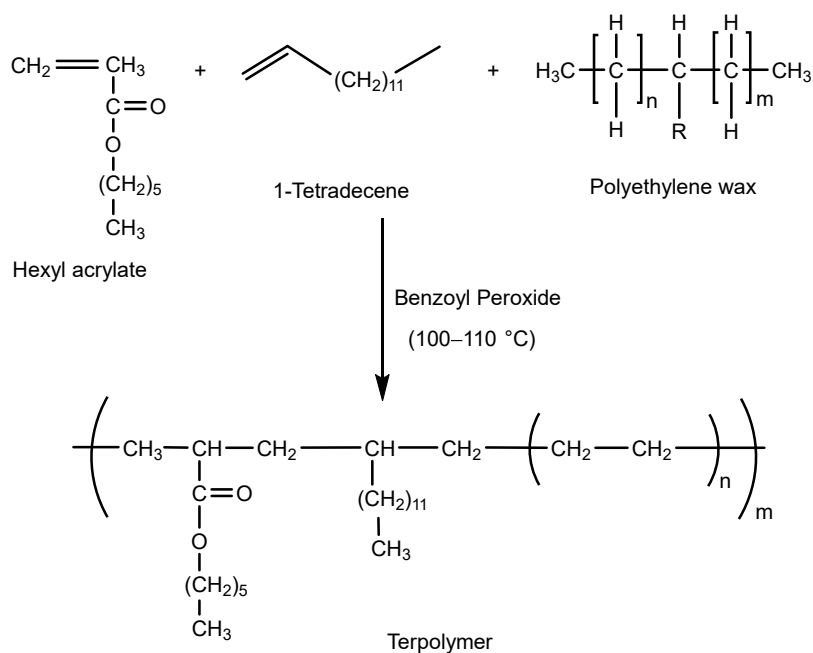| Terpolymer code	Monomers ratio (mol)			Yield
	Hexyl acrylate	1-Tetradecene	polyethylene wax	
w1	0.5	0.5	0.1	87%
w2	0.5	0.5	0.3	90%
w3	0.5	0.5	0.5	85%
w4	0.5	0.5	0.7	92%

Fig 1. Terpolymer chemical structures

before and after the addition of terpolymer additives. ASTM D-445 was used for determining the kinematic viscosity of liquid petroleum products. In this method, the time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity of the product is obtained by measuring flow time and the calibration constant of the viscometer [37]. ASTM D-2270 standard covers a procedure for calculating the VI of petroleum products, such as lubricating oils and related materials, by their kinematic viscosities at 40 and 100 °C [37]. ASTM D-97 test method outlines a procedure for determining the pour point by cooling a sample in a special apparatus and observing the temperature at which the first indication of movement of the liquid occurs when the sample container is tilted [34].

■ **RESULTS AND DISCUSSION**

FTIR analysis showed similar pattern spectrums for all four terpolymers with different peak intensities. Fig. S1 shows the FTIR spectra of the monomers and prepared terpolymer w3. The 2988 and 2862 cm^{-1} peaks are the asymmetric stretching and symmetric stretching of –CH$_2$–, respectively. Meanwhile, the peaks at 1442 and

1490 cm^{-1} represent the bending vibration of –CH [38]. A strong peak at 1732 cm^{-1} is assigned to the carbonyl (C=O) group of ester. Meanwhile, two peaks at 1149 and 1250 cm^{-1} represent C–O bond stretching of ester groups [10,39]. The absence of the C=C peak at 1680–1600 cm^{-1}, which is characteristic of monomers, confirms the terpolymer formation, and polymerization was accomplished [11,40-42].

The ^{1}H-NMR measurement was used to confirm the terpolymer's structure. All materials give similar patterns in their ^{1}H-NMR spectrum of w1, w2, w3, and w4, as shown in Fig. S2-S5. A signal at a chemical shift (δ) of 3.99–3.62 belonging to –COO–CH$_2$– of the hexyl acrylate. On the other hand, typical peak signals that correspond to a peak at δ 0.860 ppm for –CH$_3$ and a peak at δ 1.29 ppm for –CH$_2$- of alkyl chain in the monomers, and a peak at δ 2.54 for CO–CH of hexyl acrylate were also found. Also, the absence of a signal at 4–6 ppm belonging to –HC=CH– confirms the terpolymer formation [16,41].

Using TGA-DTG, the thermal stability of the synthesized terpolymers is examined, and the results are shown in Fig. 2. Terpolymer w1 shows two signals at 288.26 and 400.32 °C. Terpolymer w2 shows two signals at 150.28 and 398.73 °C. Terpolymer w3 shows two signals
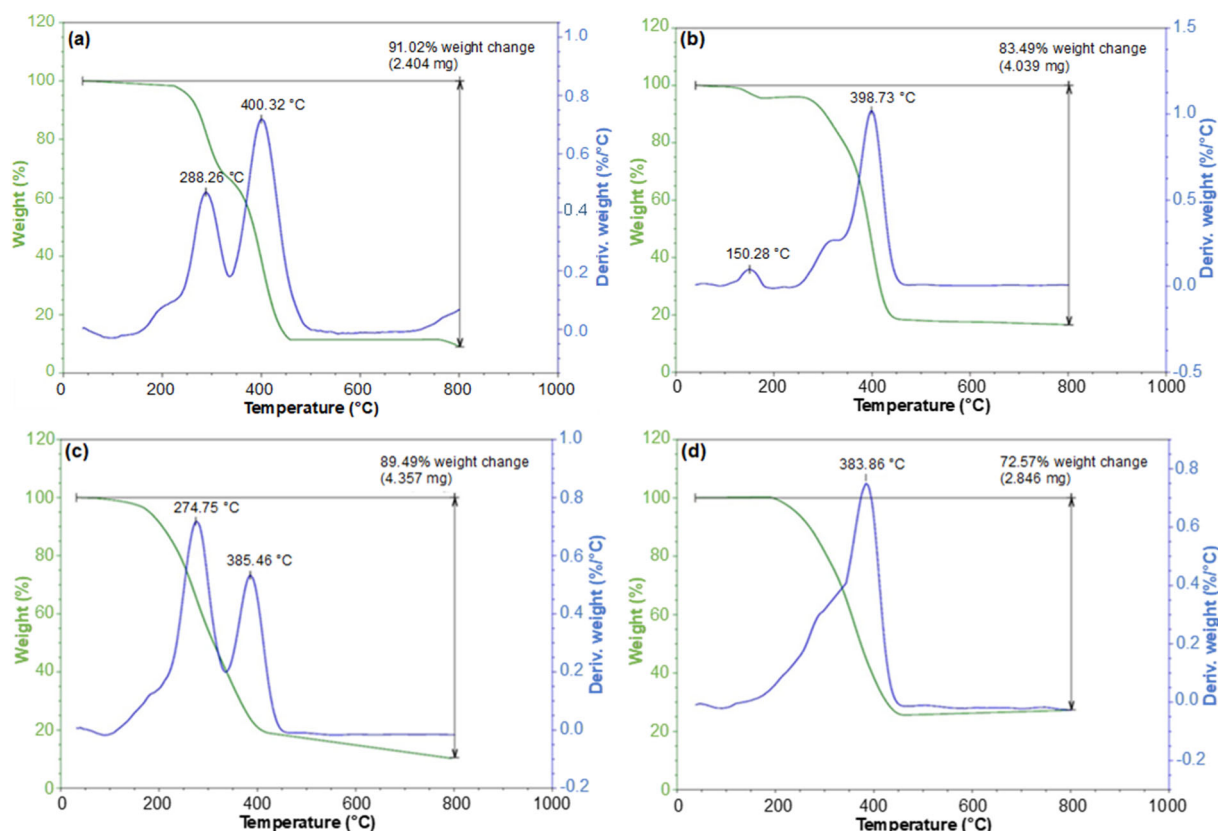
Fig 2. TGA-DTG thermogram of terpolymer additives (a) w1, (b) w2, (c) w3, and (d) w4

at 274.75 and 385.46°C. Meanwhile, terpolymer w4 shows a major signal at 383.86 °C. Fig. 2(a-d) shows that the terpolymers degrade completely at temperatures of 383–401 °C, indicating that prepared terpolymers have high thermal stability [6,10,39].

All terpolymer additives were studied with gel permeation chromatography (GPC) to determine the value of the average molecular weight (Mw) and number of average molecular weight (Mn) and study the correlation of increased (Mn) and (Mw) on the increase of molar ratio of polyethylene wax.

The value of average molecular weight (Mw) of the terpolymers is measured from their GPC profile, and the data are shown in Table 3 and Fig. 3(a-d). The data shows that the increase in polyethylene wax molar ratio causes increases in Mw. The Mw values of terpolymer w1, w2, w3, and w4 are 6781, 31622, 36307, and 40193, respectively. Meanwhile, the Mn values of terpolymer w1, w2, w3, and w4 are 3408, 4786, 1862, and 8584,

respectively, which confirms that this gradual increase in the molar ratio of polyethylene wax has achieved the required gradual increase in Mw and Mn for the terpolymer.

Evaluation as VII

The terpolymer additives were evaluated as VII using a free-additive Iraqi lubricating oil (base oil 60) by calculating VI according to the ASTM D-2270-87 standard Table 4 [43]. The kinematic viscosity of the lubricating oil with the prepared terpolymers was tested

Table 3. Terpolymers compositions and weight average molecular weight (Mw)

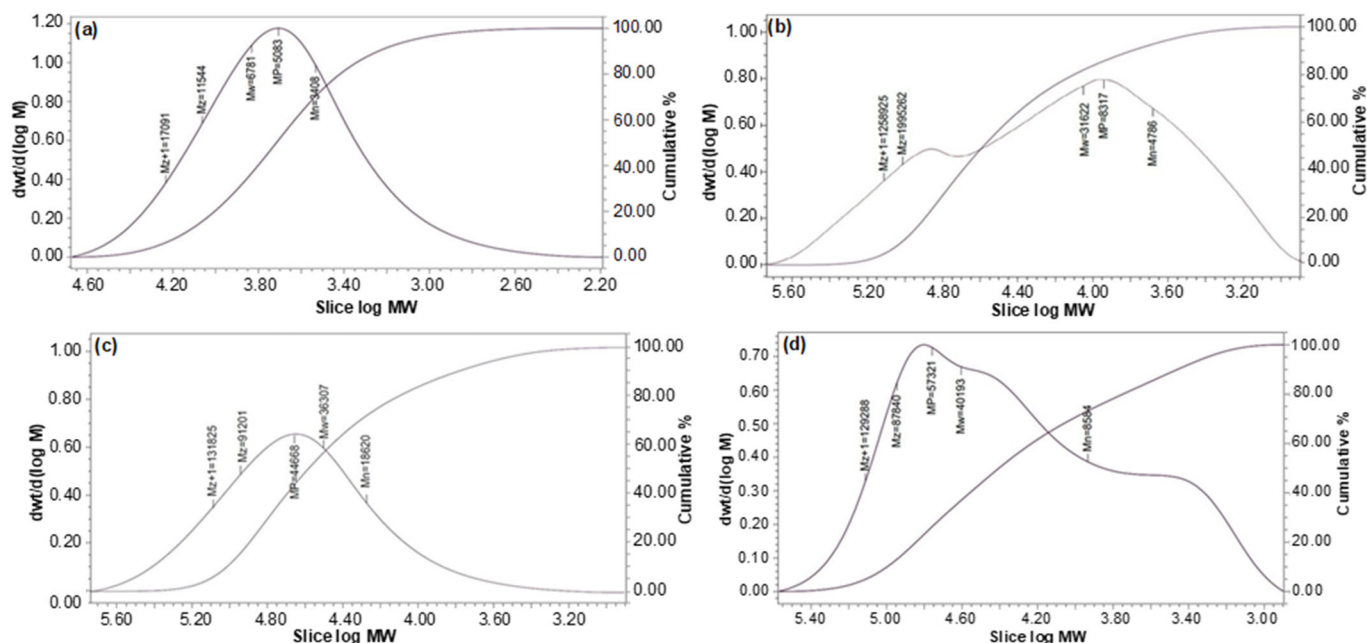| Terpolymer	Mw	Mn
w1	6,781	3,408
w2	31,622	4,786
w3	36,307	1,862
w4	40,193	8,584

Fig 3. GPC profile of terpolymer additives (a) w1, (b) w2, (c) w3, and (d) w4

at temperatures of 40 and 100 °C. A series of concentrations of 1, 2, 3, 4, and 5% by weight, were selected to examine the impact of terpolymer additives [9,15,18,44].

The Effect of the Terpolymer Concentration and Monomer Molar Ratio on the Efficiency

In order to find out how the concentration of the terpolymer additives affected VI, a range of additive concentrations, including 1, 2, 3, 4, and 5% by weight, were used; the data are given in Table 4 and Fig. 4 which show that the VI increase as the terpolymer additives concentration in the lubricating oil increases. As an example, terpolymer w1 gave the VI value of 98, 114, 123, 129, 134, and 138 for 0, 1, 2, 3, 4, and 5% weight concentration. Terpolymer w2 gave the VI value of 98, 153, 162, 174, 179, and 190 for 0, 1, 2, 3, 4, and 5% weight concentration. Terpolymer w3 gave the VI value of 98, 173, 179, 190, 192, and 206 for 0, 1, 2, 3, 4, and 5% weight concentration. On the other hand, terpolymer w4 gave the VI value of 98, 118, 126, 139, 156, and 160 for 0, 1, 2, 3, 4, and 5% weight concentration.

This behavior is consistent with the literature, attributed to an increased total volume of additives by increasing concentration [9,39,45-46]. The lubricating oil

viscosity reduces as the temperature rises; however, because of the increased solvation power, the polymer

Table 4. The VI values for the terpolymers and free additives lubricating oil

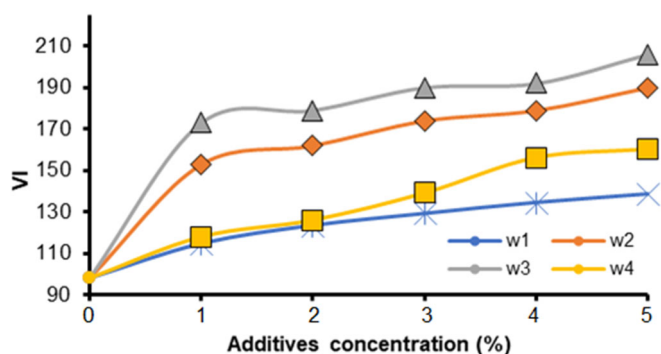| Conc. % by weight	VI			
	w1	w2	w3	w4
5	138	190	206	160
4	134	179	192	156
3	129	174	190	139
2	123	162	179	126
1	114	153	173	118
0	98	98	98	98

Fig 4. Influence of the concentration of terpolymer additives (w1, w2, w3, and w4) on the VI value of lubricating oil
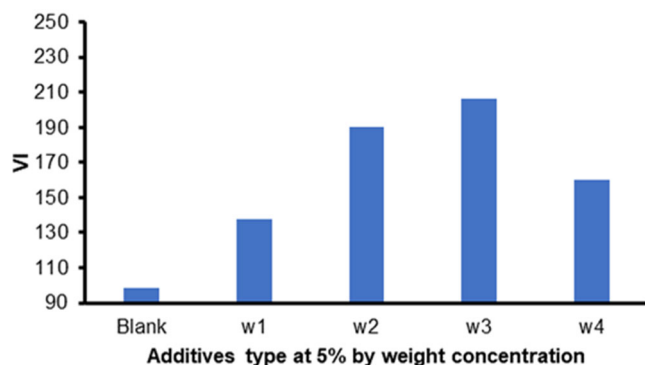
Fig 5. Influence of polyethylene wax monomer ratio in terpolymer additives on the VI value of lubricating oil

molecules change from tight coils to expanded ones due to increased interaction between the polymer chain and the solvent molecule. This increase in molecule size compensates for the loss in lubricating oil viscosity and, as a result, lowers the mixture's viscosity changes with temperature [47-48]. The oil solution's total volume of polymer increases as polymer concentration increases. Consequently, a high polymer concentration produces a higher VI value than a low polymer concentration [39,49].

When investigating the impact of the monomer ratio on the VI value of lubricating oil, as shown in Fig. 5, VI increases when the molar ratio of polyethylene wax increases. On the other hand, an increase in the molar ratio of polyethylene wax over hexyl acrylate had the opposite effect on the VI value; this could be attributed to the influence of the increase in molecular weight, which affects the effective coil size expansion, which became more complex, consequently, the decrease in the VI value [39,48]. The terpolymers w1, w2, and w3 were successful in raising VI of free additive lubricating oil, and the terpolymer w3, which contains an equal monomers ratio, gives the best efficiency in the VI value of 206 at 5 wt.% concentration compared with VI value of 98 for free additive lubricating oil.

CONCLUSION

Four terpolymers of hexyl acrylate, 1-tetradecene, and polyethylene wax have been prepared and characterized by ^{1}H-NMR, FTIR, TGA-DTG, and GPC measurements. Evaluation of these terpolymers as VII in combination with Iraqi lubricating oil (base oil 60) has revealed a substantial enhancement in efficiency by incorporating industrial waste. The improvements demonstrated a proportional relationship with terpolymer concentration, indicating the potential for optimizing performance through precise control of additive levels. The exploration of molar ratios of monomers highlighted a critical influence on terpolymer efficiency. Notably, terpolymer w3, characterized by a balanced molar ratio, exhibited the highest degree of efficacy, reaching a VI of 206 at a 5% concentration. This notable performance surpasses the baseline of free additive lubricating oil, which yielded a VI of 98. These findings present compelling insights into the sustainable utilization of industrial waste to enhance the composition of lubricants. The study contributes to advancing environmentally conscious practices in the lubricant industry and emphasizes the strategic importance of optimizing molar ratios for superior terpolymer performance. Such revelations hold considerable promise for developing more efficient and sustainable lubricating compositions, aligning with the evolving demands of industry and environmental stewardship.

CONFLICT OF 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.

AUTHOR CONTRIBUTIONS

The idea was conceived by Ziyad Tarik AL-Malki, drawing from the insights of Moayad Naeem Khalaf, who supervised the project. Ziyad Tarik AL-Malki conducted the experiment and carried out the analysis. The results were collectively discussed by all authors, contributing to the final manuscript.

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