Synthesis, Characterization and Performance Evaluation of Castor oil based additives for Lubricating oil

Abstract: The gradual reduction of worldwide crude oil stores, increasing costs of crude oil, and escalating environmental worries are critical matters with the capacity to profoundly affect human existence. Meeting these challenges necessitates researchers' dedicated focus on discovering alternative solutions and advancing the creation of a sustainable ecosystem.This research seeks to tackle these issues by creating homopolymers of castor oil (CO) and copolymers using Castor oil and n-butyl acrylate, without solvent. The process uses benzoyl peroxide (BZP) as the initiator for polymerization, and the resulting polymers are tested for their effectiveness as additives in lubricating oils. Analyzing the synthesized polymers entails utilizing spectroscopic methods like FT-IR and NMR spectroscopy for characterization purposes. Thermo-gravimetric analysis (TGA) is employed to evaluate thermal stability, while gel permeation chromatography (GPC) is utilized to ascertain molecular weight.The effectiveness of additives in enhancing viscosity index (VI) and reducing pour point (PP) is being examined through the ASTM procedure using the SN150 mineral oil standard. The disk diffusion method was utilized to evaluate the biodegradability of various polymers.The results from the experiment and the analysis of the data suggest that the copolymer shows potential as a versatile lubricating oil additive when compared to the homopolymer. Furthermore, elevating the concentration of n-butyl acrylate within the copolymer leads to heightened average molecular weight, better thermal stability, and superior effectiveness as a viscosity index improver (VII) and pour point depressant (PPD).This implies that modifying the copolymer's makeup can enhance its molecular properties and effectiveness when used as an additive in lubricating oil.

Keywords: Castor oil, Free radical polymerization, Thermal stability, Pour point depressant, Viscosity index improver, Biodegradability.

1. Introduction

The majority of lubricants commonly used today are derived from petroleum-based sources, which pose environmental risks due to their toxicity. Presently, approximately 95% of lubricants utilized rely on petroleum-derived inputs and oils[1]. Lubricating oil, comprised of various high molecular weight hydrocarbons of different chain length such as paraffinic, aromatic, and naphthenic compounds[2]. However, its effectiveness and lifespan would be significantly diminished in automotive engines without the inclusion of additives which are polymeric in nature. Additives are comprised of a non-polar hydrocarbon chain along with a polar head group. These two components in additives are responsible for its distinct solubility in oil, it often take the form of inverse micelles when dispersed in a colloidal solution[3]. Incorporating additives into lubricating oil is crucial for ensuring the smooth operation of modern engines. Lubricants, serve various vital roles such as maintaining separation between moving parts, minimizing friction, shielding against wear, facilitating heat transfer, preventing rust and corrosion, and acting as antioxidants[4], viscosity index improvers [5] and detergents/dispersants[6]. There is abundant literature available regarding the utilization of altered vegetable oils as additives in lubricating oils[7]. Petroleum-based lubricants performed well but pose environmental concerns due to their eco-toxicity and lack of biodegradability. Furthermore, the finite resources of the commonly utilized lubricants are diminishing, leading to a gradual rise in their expenses[8]. As a result, there's a growing focus on exploring vegetable oils as alternatives for base stocks or additives, driven by their biodegradability, antiwear characteristics [9], minimal volatility[10], pour-point depressants and high viscosity index[11], improved performance of extreme pressure additives[12]. However, because of certain limitations such as inadequate fluidity in cold conditions (known as pour point), insufficient oxidative stability, subpar resistance to biological degradation, and limited hydrolytic stability[13] using vegetable oils directly as a base stock for lubricants does not yield satisfactory performance. However, these constraints can be lessened through chemical modification methods such as epoxidation, metathesis, polymerization etc., applied to the oleochemicals found in vegetable oils[14,15]. Methacrylates are commonly employed to lower the pour point and modify the viscosity of lubricating oils[16]. In recent years, researchers have prioritized the development of eco-friendly lubricants and additives to support environmental sustainability and promote a greener approach [17].

Castor oil *(Ricinuscommunis)* has been considered for polymerization due to its widespread availability for trade, affordability, and suitable levels of unsaturation. Castor oil is made up of triglycerides of fatty acidsobtained from the seeds of the castor bean. These fatty acids consist of various triglyceride most of which are unsaturated(-C=C-) leading to reduced thermal and oxidative stability[18]. Ricinoleic acid is C-18 fatty acid characterized by a double bond at C-9 and a hydroxyl group at C-12 constituting 90% of its composition [19]. This abundance of ricinoleic acid is the key factor contributing to its high value and adaptable uses across diverse fields. Castor oil finds extensive applications across various significant sectors including lubrication, coatings, rubber, pharmaceuticals predominantly employed as a remedy for constipation, glues, cosmetics, and more[20]. Combining castor oil with acrylates through copolymerization not only boosts the polarity of the unit but also greatly improves its thermal and mechanical stability[21].The paper conducts polymer synthesis, subsequent characterization and assessment of performance.

This research introduces a method for creating a new lubricant blend with improved characteristics in its additives for flexibility. So, in this study, we've created a homopolymer derived from castor oil and a copolymer with n-butyl acrylate in varying proportions to develop lubricant additives that are both thermally stable, economical, and environmentally friendly. n-butyl acrylate was selected for its ability to improve the thermal stability of lubricants. The polymeric additives were then assessed for their effectiveness as viscosity index improvers and pour point depressants using the appropriate ASTM methods.

2. Experimental section

2.1. Materials

Castor oil with a high unsaturation level of 96.7% was acquired from a local grocery store[22]. Table 1 presents the fatty acids composition in castor oil, while Figure 1 illustrates the structure of its primary component, which is the triglyceride of fatty acids[23]. Benzoyl peroxide, obtained from LOBA Chemie in India with a purity of 98%, was used after undergoing recrystallization using a blend of chloroform and methanol ($CHCl_3$-MeOH. n-butyl acrylate was obtained from Sisco Research Laboratories Pvt. Ltd. Base oil SN150 was collected from Indian Oil Corporation Limited (IOCL), Dhakuria, West Bengal, India. The fungal samples were collected from the Microbiology Department at North Bengal University in West Bengal, India.

Table 1. Fatty acid composition of Castor oil[22].

Fatty acid	Molecular Formula	Percentage (%)
Ricinoleic(Unsaturated)	$C_{18}H_{34}O_3$	87.7-90.4
Linoleic (Unsaturated)	$C_{18}H_{34}O_3$	4.2-4.7
Linolenic (Unsaturated)	$C_{18}H_{34}O_3$	0.3-0.7
Oleic (Unsaturated)	$C_{18}H_{34}O_3$	2.2-3.3
Stearic (Saturated)	$C_{18}H_{34}O_3$	0.7-1.0
Palmitic (Saturated)	$C_{18}H_{34}O_3$	0.8-1.1

2.2. Synthesis of the polymers (Homopolymer and copolymer)

The synthesis of a homopolymer derived from castor oil and its copolymer with n-butyl acrylate was conducted in a three-necked round-bottom flask equipped with a mechanical stirrer, condenser, thermometer, and a nitrogen inlet without any solvent.The mixture was stirred at 50 °C for 30 min to get a homogeneous solution followed by the addition of Benzoyl peroxide (BZP 0.5% w/w) which was used as an initiator.The reaction proceeded at 90°C for 6 hours via free radical polymerization. After the reaction time, the reaction mixture was transferred into methanol while being stirred to stop the polymerization process and cause the polymer to precipitate. The polymers were then purified by multiple rounds of precipitation from their hexane solution using methanol, and subsequently dried under vacuum at 40 °C[24,25]. The steps involved in the synthesis of polymer are given below:

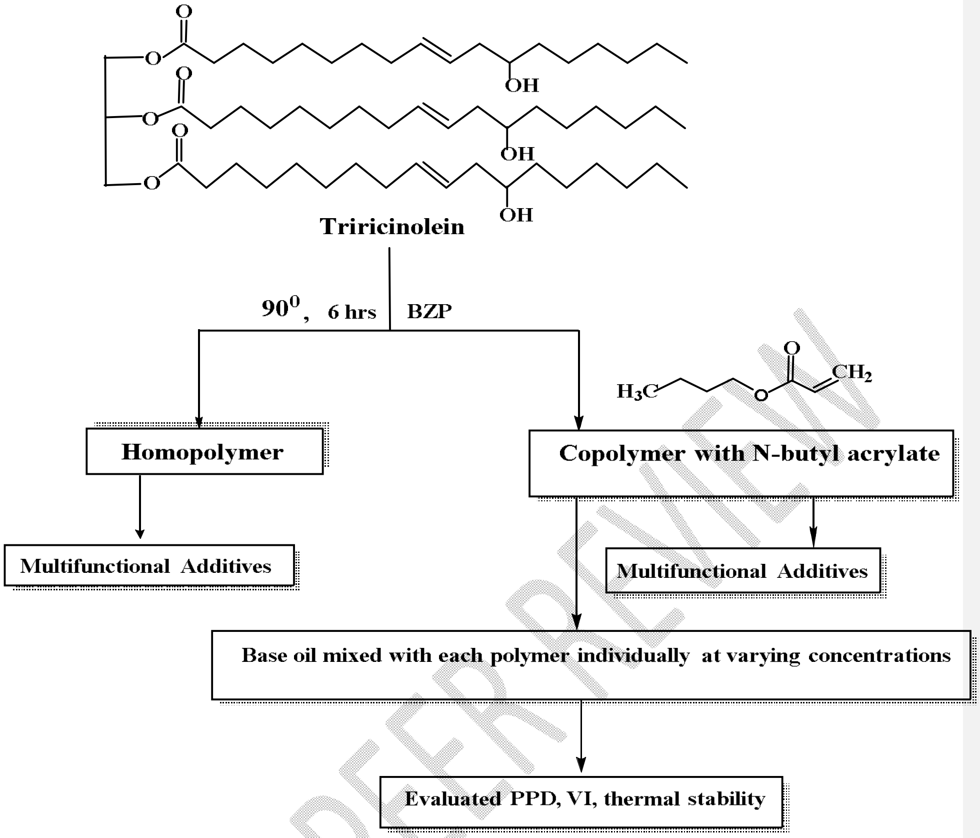

Fig. 1 Polymerization of Castor oil and n-butyl Acrylate

2.3. Determination of average molecular weight by GPC

The SEC-GPC device was used to determine the number-average molecular weight (Mn), weight-average molecular weight (Mw), and polydispersity index (PDI) of the synthesized polymers. The GPC system (polystyrene calibration) was equipped with a 2414 refractive index detector, Waters 515 HPLC pump and a 717 plus auto sampler, 2489 UV/Visible detection. HPLC grade THF was used as an eluent for the instrument at a flow rate of 1.0 mL/min at 30° C. Table 3 shows the average molecular weight values as determined by GPC[26].

2.4. Spectroscopic analysis

The polymers were analyzed using BRUKER AVANCE II 400 MHz FT-NMR instruments. Infrared (IR) spectra were obtained using a Shimadzu FT-IR 8300 with 0.1

mm KBr cells at room temperature, covering the range of 400 to 4000 cm^{-1}. ^1H NMR analysis was conducted with 5 mm BBO probes, utilizing $CDCl_3$ solvent as the reference.

2.5. Thermogravimetric analysis (TGA)

The thermo-oxidative stabilities of the polymers were assessed using a Shimadzu TGA-50 thermo - gravimetric analyzer in an air environment, employing an alumina crucible. Heating was conducted at a rate of 10º C/min, and the percentage of weight loss (PWL) of the polymers as temperature increased was determined.

3. Performance evaluation

3.1. Evaluation of viscosity index

Viscosity index (VI) is a unit less qualitative parameter that signifies how much a base oil's viscosity changes with the change in temperature. A higher VI suggests that the viscosity remains more stable despite temperature changes, while a lower VI indicates greater variation in viscosity with temperature.The effectiveness of an additive like a viscosity index improver (VII) is evaluated based on how much it enhances the viscosity index (VI) of the base oils when added to them.The ASTM D2270-10 method was used to calculate by measuring the kinematic viscosity of base oils at both 40^o C and 100°C subsequent to blending additives at various concentration levels.Various concentrations of additives, ranging from 1% to 4% (w/w) were mixed with the lubricating oil to investigate how the concentration of additives impacts the viscosity index[27].

3.2. Evaluation of Pour point of the additives in Lube oil

At colder temperatures, lubricants solidify into a gel-like consistency, preventing their flow. The temperature at which this flow stops is known as the pour point (PP) of the lubricant. This occurs because of the formation of paraffinic wax crystals at lower temperatures within the lubricant, which can result in engine component failure if not addressed.The pour point of the lubricant blend was analyzed using the ASTM D 97-09 standard procedure with a cloud and pour point tester known as the WIL-471 model from India. A quality pour point depressant additive effectively reduces the pour point of the lubricant mixture to a significant extent. Different amounts of both lubricant and additives, ranging from 1% to 4% (w/w) were employed to examine how the pour point of lubricating oil changes with the concentration of additives[28].

4. Biodegradability Test

One key benefit of utilizing vegetable-derived additives instead of synthetic acrylates lies in their remarkable ability to degrade naturally, highlighting a significant difference between vegetable-based oils and mineral oils. Unlike mineral oils, vegetable-based oils easily break down, especially in anaerobic environments[29].Ultimate biodegradation refers to the full breakdown of a substance by microorganisms into carbon dioxide, water, mineral salts, and microbial biomass. Numerous methods have been developed to measure biodegradability, with this study using the disc diffusion technique to assess how polymers are broken down by fungal pathogen [30].

Disc Diffusion (DD) Method

The disc diffusion technique involves preparing culture media for fungal strains and examining how well additives degrade when exposed to fungal pathogens. The prepared additives were subjected to biodegradation testing against fungal pathogens such as *Alternaria alternata* (AA) using a bacteriological incubator. A culture medium was created by blending agar powder, potato extract, and dextrose in a ratio of 1:10:1 (weight/weight). Petri dishes containing 2 grams of this medium, along with 1.0 grams of various polymeric additives, were incubated at 310 Kelvin for a month to observe their interaction with different types of fungal pathogens. The presence of fungal growth was established when the colour shifted from yellow to blackish. Following a 30-day period, the additive samples were extracted from the fungal environment, treated with chloroform, purified, and then dried. Subsequently, the weight reduction of each sample was determined[31].

5. Results and discussions

5.1. Molecular weight data analysis

The Gel Permeation Chromatography(GPC) was utilized to determine the weight average (M_w) and number average (M_n) molecular weights of the synthesized polymers (P-1 to P-4) at room temperature in THF, as indicated in Table 2. The analysis of the experimental findings indicates that among the four polymers, P-4 demonstrates the greatest molecular weight. Furthermore, there is a noticeable relationship between the molecular weight and the proportion of n-butyl acrylate in the castor oil structure: as the percentage of n-butyl

Comment [R1]: Is it necessary to indicate in the text what are the differences in the reaction conditions to synthesize P-1 or P-4? Why were 4 polymers synthesized?

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acrylate rises, so does the molecular weight[32].This underscores the considerable impact that n-butyl acrylate has on the process of polymerization.

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Table 2. Molecular weight of the prepared polymer (M_n, M_w and PDI value)

Polymer sample	M_n	M_w	PDI
P-1	2277	2729	1.198
P-2	2770	3169	1.143
P-3	3790	4264	1.125
P-4	5750	8852	1.539

P-1:100% of CO; P-2: 97.5% of CO + 2.5% of n-butyl acrylate; P-3:95% of CO + 5% of n-butyl acrylate; P-4: 92.5% of CO +7.5% of n-butyl acrylate; M_n: Number average molecular weight; M_w: Weight average molecular weight; PDI: Polydispersity index.

5.2. **Spectroscopic data analysis**

The homopolymer and copolymer derived from castor oil both displayed an infrared absorption peak at 1739.40 cm^{-1} and 1736.20 cm^{-1}, indicating the presence of the ester carbonyl group[33]. The peaks ranging from 1165.15 cm^{-1} –to 1025.8 cm^{-1} in the homopolymer and 1164.30 cm^{-1} to 1117.2 cm^{-1} in the copolymer were associated with the stretching vibration of the ester C-O bonds. In the homopolymer, the peaks observed around 1457.43 cm^{-1}, 1270.08 cm^{-1}, and 735 cm^{-1} correspond to the bending vibrations of the C-H bonds in the methylene and methyl groups of castor oil. Similarly, in the copolymer, peaks at approximately 1458.02 cm^{-1}, 1269.37 cm^{-1}, and 713.38 cm^{-1} indicate the same bending vibrations for these groups.The wide IR transmission observed at 3442.63 cm^{-1} and 3457.16 cm^{-1} corresponds to the O-H stretching associated with the alcoholic group found in ricinoleic acid, which is a component of castor oil. The absorption bands detected between 2925.42 cm^{-1} and 2855 cm^{-1} in the homopolymer, and from 2925.54 cm^{-1} to 2855 cm^{-1} in the copolymer, were attributed to the stretching of C–H bonds. The absence of peaks between 1600 cm^{-1} and 1680 cm^{-1} suggests there is no carbon bonds characteristic of olefins in the polymers, confirming the successful formation of the intended product[26].

Comment [R4]: Where is the IR spectrum?

The ^{1}H NMR spectra for both the homopolymer of castor oil and its copolymer with n-butyl acrylate show peaks between 2.3 ppm and 2.5 ppm. These peaks indicate the presence of protons linked to the $–OCH_2$ groups in Castor oil and n-butyl acrylate.

Furthermore, peaks detected between 0.6 ppm and 1.5 ppm signifies the presence of methyl protons, while those between 1.8 ppm and 2.5 ppm indicate methylene protons from Castor oil and n-butyl acrylate. In the copolymer, the presence of protons from –$COOCH_2$ groups is suggested by peaks appearing between 3.5 ppm and 4.5 ppm. The formation of the homopolymer was indicated by the absence of peaks in the unsaturation region between 4.5 ppm and 5.2 ppm. The creation of the copolymer from Castor oil and n-butyl acrylate was verified by the absence of peaks in the 5.8 ppm to 7.0 ppm range, suggesting successful polymerization. These findings align with previously reported results[29].

Comment [R5]: Where are the NMR spectrum?

In the ^{13}C NMR spectra for both the homopolymer and copolymer derived from castor oil, the ester carbonyl group appears in the range of 170 ppm to 177 ppm. Additionally, the carbon atoms of the –$OOCH_2$ group in castor oil are observed between 61 ppm and 70 ppm. Furthermore, the lack of peaks within the 135 ppm to 160 ppm range confirms the successful polymerization process. These findings align with previously reported results [32].

5.3. Thermogravimetric analysis (TGA)

The thermal stability of the polymers was assessed using their TGA values. It's clear from the result that the homopolymer P-1 exhibits lower thermal stability compared to the other copolymers like P-2, as evidenced by its higher rate of thermal degradation. As the concentration of Castor oil decreases in the feed, the thermal stability also increases. The primary degradation of homopolymer P-1 occurs at 250° C, resulting in a 25% weight reduction, while copolymer P-2 experiences this at 300° C. The incorporation of Castor oil with n-butyl acrylate in copolymerization enhances the overall thermal stability of the polymer. So, incorporating n-butyl acrylate through copolymerization stands as a crucial factor in improving the polymer's thermal stability, a key requirement for it to serve as an additive in lubricating oil.

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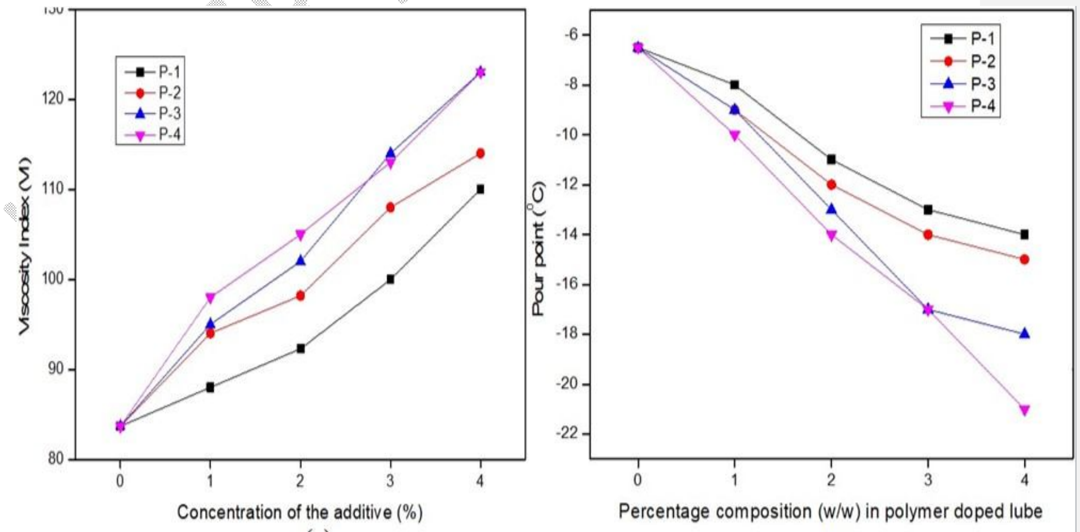
5.4. Analysis of Viscosity index (VI) value

Viscosity index (VI) values of the base oils combined with the prepared additives are depicted in Fig.2. The findings show that the viscosity index (VI) values are elevated in the blends/lubricants when contrasted with the base oils without additives. Furthermore, these values tend to rise as the concentration of the blended additives increases. All polymers exhibited impressive outcomes, with copolymers demonstrating superior performance as viscosity index improvers (VII) compared to homopolymer. Among copolymers, P-4 showed the highest enhancement in viscosity index (VI). A higher VI value indicates that the newly created additive molecules can better maintain viscosity stability across a broader temperature range in mineral base stocks and it may be due to increased of volume in polymer micelles [34]. As a result, increased concentrations of the additive will cause the viscosity index to rise.

5.5. Analysis of pour point values

Based on the pour point information given, it's evident that all the synthesized polymers effectively serve as excellent pour point depressants for the specified base oil. The outcomes of the experiments are detailed in Fig.2. PPDs typically contain a paraffinic component that resembles wax, which bonds with the waxy elements in oil, essentially integrating with them. The presence of polar components in PPDs restricts the extent to which this bonding can occur[35]. Adding acrylate co-monomers to the castor oil backbone increases the polarity of the molecule. As a result, the copolymers exhibit greater efficacy in inhibiting the crystallization of paraffin wax within mineral oil and the formation of a gel-like structure at lower temperatures (pour point).The P-4 additives have demonstrated superior performance in terms of PPD compared to other polymers,likely attributed to its higher polydispersity index (PDI) value[36].



(a) (b)

Fig. 2. (a) Viscosity index (VI) and (b) Pour point (PP) of the prepared polymers in lube oil

5.6. Biodegradability test results

The results of biodegradability test carried by disc diffusion technique are depicted in Fig. 3. The test results show that the breakdown of P-1, homopolymer exhibits the greatest extent. It was observed that the degradation of other copolymers also occurs notably, with the P-4 copolymer experiencing the least degradation. After 30 days period, the weight loss of P-1 and P-4 was found 90% and 39% respectively.

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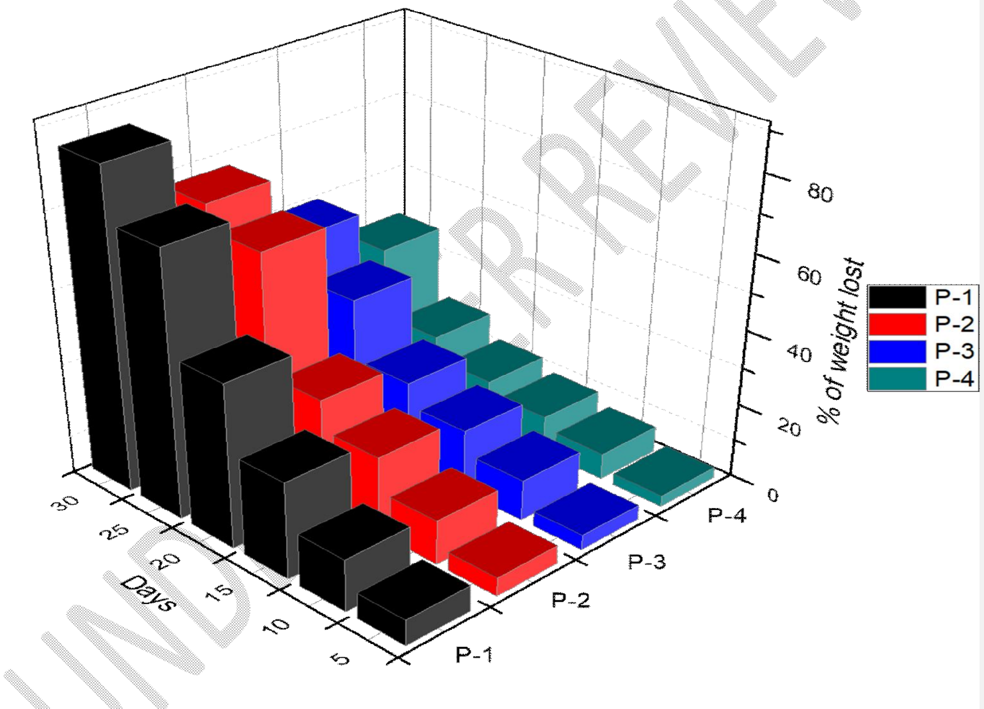

Fig. 3. Biodegradability test of the lubricant composition

6. Conclusions

The above study reveals that copolymers are more efficient than homopolymers. Both the homopolymer of castor oil and its copolymer with n-butyl acrylate demonstrated superior performance as viscosity index (VI) improvers and pour point depressant additives for base oil. Thermal stability, average molecular weight and performance as viscosity index improvers (VII) all improve with a higher percentage of n-butyl acrylate in the copolymers. The effectiveness of the additives as pour point depressants (PPD) improves with higher additive concentrations, reaching optimal performance at a 4% concentration. Homopolymers exhibit greater biodegradability compared to copolymers, and the biodegradability decreases as the percentage of n-butyl acrylate in the copolymers increases. Consequently, the aforementioned study presents a viable method for developing an eco-friendly lubricant composition.

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