

# **Preparation and characterization of polymeric dispersants based on vegetable oils for printing ink application**

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## **Abstract**

In this work, a novel vegetable oil-based polymers were prepared by epoxidation of soybean oil (SBO) and castor oil (CO) followed by ring opening reaction of epoxidized oil with polyether amine and poly propylene glycol. The prepared polymers were characterized by FTIR and GPC. The properties of vegetable oils and epoxidized vegetable oil (EVO) were studied. The prepared polymers were employed as novel polymeric dispersants for pigment dispersion in solvent based printing ink application. The mechanical and optical properties of prepared ink were studied. The net technical properties of the new ink formulations are relatively comparable to the prepared printing ink from standard polymeric dispersant. The polymeric dispersant 2 (PD2) and polymeric dispersant 4 (PD4) gave the best optical and mechanical properties among the prepared polymers.

**Keywords:** Vegetable oils, Printing inks, Polymeric dispersants, Pigment, Castor oil.

## **1. Introduction**

The manufacture of printing ink is a technologically advanced, highly specialized and complex process [Pal L and Fleming P (2006)]. Preparation of stable, homogeneous and fine dispersion of pigment is not facile because its prone to aggregate. The dispersion of pigment strongly affect the optical properties such as color strength, transparency, gloss and mechanical properties such as adhesion of printed ink film [Nsib F et al (2006); Kuo K et al (2008); Tryznowska ZZ (2016)]. Stabilization of pigment dispersion usually results from adsorption of dispersing agent molecules from solution onto the particle surface of the pigment creating repulsive forces between the particle in suspension either through electrostatic repulsion or from steric prevention of coagulation.

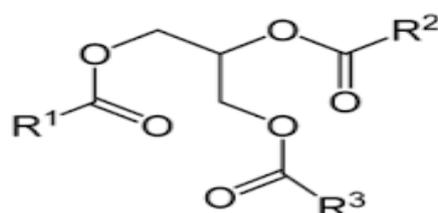
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The degree of dispersion of a particle suspension may be defined as the extent to which the individual powder particles become separated from one another in the liquid medium [Reuter E et al (1999); Nsib F et al (2006); Wang J et al (2006);].

Polymeric pigment dispersants are copolymers with pigment affinic “anchoring groups” and soluble polymeric chains [Schofield JD and Calbo LJ (1992); Spinelli HJ (1998)]. The polymeric dispersants are used to obtain homogeneous dispersion of the pigment in the liquid phase that leads to weak viscosity and allows high pigment loading and high tinting strength [Clarke JB (1997); Schofield JD (2002); Lokhande GP and Jagtap RN (2016)].

Health-related issues, stringent environmental protection policies, search for cost-effective and alternative materials and the quest for renewability, sustainability and high-performance materials for technical applications have led to intense research in the production of renewable polymers from plant seed oils and shift in focus from the petrochemical based polymers [Salih N et al (2011); Saithai P et al (2013); Adekunle KF (2015)]. Vegetable oils are a part of large family of chemical compounds known as fats or lipids which are made up predominantly of triesters of glycerol with fatty acids which can be processed into high value oleochemicals for various industries [Abdullah BM and Salimon J (2010)]. The general structure of vegetable oils is shown in *Figure 1* which is triglycerides comprise three fatty acids joined by a glycerol center [Sudha GS et al (2015)].

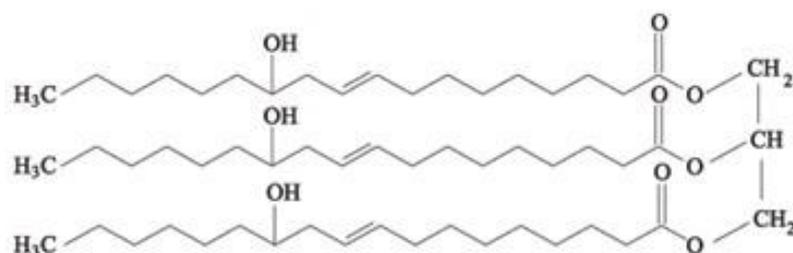


**Figure. 1 Triglyceride chain containing three fatty acids by a glycerol center.**

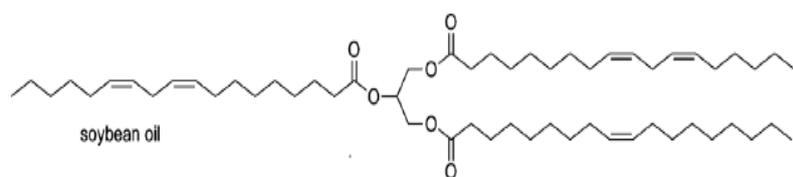
The fatty acids of vegetable oils may be saturated and unsaturated fatty acids. Castor oil, cottonseed oil, linseed oil, jatropha oil, rapeseed oil and soybean oil are examples on vegetable oils [Vlček T and Petrović ZS (2006); Sudha GS et al (2015)]. The unsaturation present (double bond) in vegetable oils can be chemically modified to form epoxidized vegetable oils [Vlček T and Petrović ZS (2006); Zhan G et al (2008); Balo F (2011)]. Epoxidation of double bond has been studied in many papers [Meyer P et al (2008); Milchert E et al (2010); Mohammad M et al (2011); Saremi K et al (2012); Mushtaq M et al (2013)]. Epoxidation is generally performed using organic peracids formed in situ via the attack of H<sub>2</sub>O<sub>2</sub> on a carboxylic acid in aqueous solution [Milchert E et al (2010)]. Due to the high reactivity of the oxirane ring, epoxides can also be used for the synthesis of

chemicals like olefinic, carbonyl compounds, alcohols, alkanolamines, glycols and polymers like polyurethanes, epoxy resin, polyesters [Dinda S et al (2008); Mohammad M et al (2011); Mushtaq M et al (2013)]. Castor oil, soybean oil are the target of this work. Castor oil is obtained from the seeds of the castor oil plant *Ricinus*. It consists of about 90% ricinoleic acid (12-hydroxy cis-9-octadecenoic acid), due to its unique structure, having fatty acids with many hydroxyl groups, makes it a very useful for industrial purpose like cosmetics, paints, adhesives, plastics, rubbers, and pharmaceuticals [Can E et al (2006); Kim HM et al (2010); Alaa MA et al (2014)].

Soybean oil is one of the most studied oils because of its ecofriendly nature and cost effectiveness. In soybean oil the major fatty acid is linoleic acid. Thus it was greatly utilized in versatile industrial fields including: coatings, printing inks, adhesives, lubricants and plastics [Eren T et al (2003); Eren T and Küsefoğlu SH (2004); Zhu J et al (2004); Holser RA (2008); Takahashi T et al (2008); Biswas A et al (2008); Lubguban AA et al (2009); Liu W et al (2016);]. *Figures 2-3* represent the structures of castor oil and soybean oil, respectively.



**Figure. 2 Major triglyceride of castor oil.**



**Figure. 3 Major triglyceride of soybean oil.**

Among the various reactions of oxiranes aminolysis is a classical route to  $\beta$ -amino alcohols formation, an important class of compounds with pharmaceutical and biological properties [Aug J and Leroy F (1996)]. Many compounds were prepared by the aminolysis of epoxides with amines under basic or acidic catalysts in organic solvents [Curini M et al (2001); Philippe C et al (2009); Stropoli SJ and Elrod MJ (2015)]. In the present study, we report the synthesis of ecofriendly polymeric dispersant for pigmented ink application by

epoxidation of soybean oil, castor oil followed by ring opening using polyether amine and polypropylene glycol.

## 2. Materials and Methods

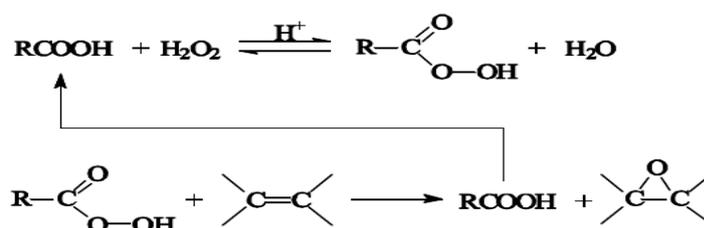
### 2.1. Materials

Chemicals were obtained as follows: polyether amine Jeffamine M-2005 (MW= 2000 g/mol) (Huntsman-Belgium), polypropylene glycol (Invista Specialty Chemicals), castor oil (CO) and commercial cooking-grade soybean oil (SBO) (a local market), ethyl acetate and methanol (Petrochem-KSA), hydrogen peroxide 50% (Piochem), zinc chloride, magnesium sulfate and *p*-toluene sulfonic acid (Oxford), formic acid, acetic acid, sodium chloride, sodium carbonate (ADWIC-Egypt).

### 2.2. Preparation of polymeric Dispersants

#### 2.2.1. Epoxidation of vegetable oil

Vegetable oils and acetic acid (in case of castor oil) or formic acid (in case of soybean oil) were mixed in a round bottom flask and stirred at 550 rpm under controlled temperature in a water bath with a temperature of  $50 \pm 2$  °C. To start the epoxidation, hydrogen peroxide solution (50%) was added drop wisely into the mixture during the first 2 h of the reaction. The molar ratio of carbon double bonds to hydrogen peroxide (C=C:H<sub>2</sub>O<sub>2</sub>) was 1:1.7. After charging H<sub>2</sub>O<sub>2</sub> was completed, the reaction continued under mixing and controlling the temperature at 50 °C for a further 5 h then the mixture was cooled to room temperature. To purify the epoxidized oil, it was poured into a separating funnel and the oil layer was washed successively with 5% sodium carbonate solution and 5% sodium chloride solution, respectively. Ethyl acetate was used to enhance the separation of the oil product from water phase. The water and solvent were then distilled off under a rotary vacuum evaporator. The oil phase was further dried above anhydrous magnesium sulfate and then filtered. Epoxidation reaction is represented in *Figure 4*.



**Figure 4: Reactions of epoxidation of oils with peracid formed in situ.**

A series of polymeric dispersants were prepared based on two different types of epoxidized vegetable oils (Epoxidized soybean oil, Epoxidized castor oil) by conventional

epoxidation method followed by ring opening using polyether amine and polypropylene glycol.

### **2.2.2. Preparation of polymeric dispersant 1 (PD1)**

PD1 was prepared by ring opening of epoxidized soybean oil in which epoxidized oil was mixed with methanol in presence of *p*-toluene sulfonic acid as a catalyst and ethyl acetate as a solvent at 50° C for partially ring opening of epoxidized oil. The partially ring opened epoxidized oil was cool down, Jeffamine M-2005 was added and the temperature was raised to 75-80° C in presence of ZnCl<sub>2</sub> as a catalyst. The reaction mixture was left for 4 h then cooled to room temperature. The prepared polymer was purified by filtration of solid catalyst. The excess ethyl acetate was distilled by vacuum distillation to obtain product of approximately 100% solid content.

### **2.2.3. Preparation of polymeric dispersant 2 (PD2)**

PD2 was prepared using the previous method for preparation of PD1, but using jeffamine only for the ring opening of the epoxidized soybean oil.

### **2.2.4. Preparation of polymeric dispersant 3 (PD3)**

PD3 was prepared by ring opening method of epoxidized soybean oil using polypropylene glycol where epoxidized soybean oil and polypropylene glycol were mixed in round bottom flask in the presence of *p*-toluene sulfonic acid as a catalyst and at temperature of 110° C for 8 h. Ethyl acetate was added to the reaction mixture as a solvent. The prepared polymer was filtered and washed by water to remove the catalyst. The excess water and ethyl acetate was distilled by vacuum distillation to obtain product of approximately 100% solid content.

### **2.2.5. Preparation of polymeric dispersant 4 (PD4)**

PD4 was prepared by ring opening of epoxidized castor oil (ECO) where ECO was mixed with Jeffamine M-2005 in a round bottom flask in the presence of zinc chloride as a catalyst and at temperature 60–70° C for 4 h. Ethyl acetate was added to the reaction mixture as a solvent. The prepared polymer was purified by filtration of solid catalyst. The excess ethyl acetate was distilled by vacuum distillation to obtain product of approximately 100% solid content.

## **2.3. Characterization**

### **2.3.1 Analytical methods**

#### **2.3.1.1 Acid value**

Acid value was determined according to DIN EN ISO 660 (Animal and vegetable fats and oils–determination of acid value and acidity).

### 2.3.1.2 Iodine value

Iodine value was determined according to DIN EN ISO 3961 (Animal and vegetable fats and oils-determination of iodine value).

### 2.3.1.3 FTIR analysis

FTIR spectra were conducted using Bruker FTIR analyzer; ALPHA-Platinum FT-IR Spectrometer with ATR Platinum–Diamond sampling module from 4000 to 400  $\text{cm}^{-1}$ .

### 2.3.1.4 Molecular weights determination

The molecular weights were determined using gel permeation chromatography (GPC) Agilent model 1515 pump system equipped with 1260 infinity refractive index detector and using tetra hydro furan (THF) as eluent, operating with a flow rate of 1.00 mL/min at 35° C. Column PL-gel 3  $\mu\text{m}$  Mixed E 300 7.5 mm covering a molecular weight range of 600–400,000 mg/g was used and calibrated using polystyrene standards.

### 2.3.1.5 Water content

Water content was determined using the Karl Fischer Titrator model Metrohm 870 Titrino plus according to (DIN EN ISO 8534).

### 2.3.1.6 Solubility in common solvents

The test was performed for the prepared polymeric dispersant in which a 50:50 solution was prepared from the polymer and common solvents are ethyl acetate, ethanol, isopropanol and toluene.

## 2.3.2. Ink characterization

The formulated ink was tested using the following techniques:

### 2.3.2.1. Printed film thickness

The printing ink was formulated according to the formulation given in **Table 1**. Then it was applied on polypropylene film (thickness 25  $\mu$ ) using hand coater. The film thickness was measured using KAFER micrometer and was found to be 3  $\mu$  for all ink samples.

**Table 1: Formulation of printing ink**

<b>Component</b>	<b>Weight %</b>
<b>Nitrocellulose varnish</b>	30 gm
<b>Pigment Cyan 15:3</b>	12 gm
<b>Plasticizer</b>	3.5 gm
<b>Polymeric dispersant (as100% solid)</b>	1.5 gm
<b>Ketonic resin</b>	5 gm
<b>Ethanol</b>	15 gm
<b>Ethyl acetate</b>	33 gm
<b>Total weight</b>	<b>100 gm</b>

### 2.3.2.2. Curling

The curling of the polypropylene printed ink film was carried out according to (ASTM D4825-97).

### 2.3.2.3. Adhesion

Adhesion was measured according to (ASTM D2252-03) and examined visually for the detached ink from the printed film.

### 2.3.2.4. Stability test

Stability test of ink was performed according to (ASTM D 1849–95).

### 2.3.2.5. X-rite measurements (Relative color strength- $\Delta E$ –Transparency)

Printing ink strength, lightness and shade were measured using EXACT–PANTONE X-rite spectrophotometer, where;  $\Delta E$  represents the differences between samples and standard in these three parameters lightness (l), red/green axis (a), yellow/blue axis (b).

### 2.3.2.6. Gloss

The gloss of the printed film was measured on the printed polypropylene film using BIUGED BGD 514 (60°) gloss meter.

## 3. Results and discussion

### 3.1. Fatty acid composition of oils

Approximately fatty acid distribution of soybean and castor oil is shown in **Table 2**.

**Table 2: Fatty acid distribution of soybean oil and castor oil**

Fatty acid %	Palmitic	Stearic	Oleic	Linoleic	Linolenic	Ricinoleic	other
Soybean	11	4	26	52	7	-	-
Castor	1	1	3	4.2	0.3	89.5	1

### 3.2 Acid value and iodine value

The characteristics features of oils are shown in **Table 3**

**Table 3: Characterization of used oils**

Oil	Acid value mg NaOH/g	Iodine value gm I <sub>2</sub> /100gm
Soybean oil	1.42	125.8
Castor oil	1.98	84.6

### 3.3. Properties of polymeric dispersant

The characteristics feature of polymeric dispersants are shown in **Table 4**.

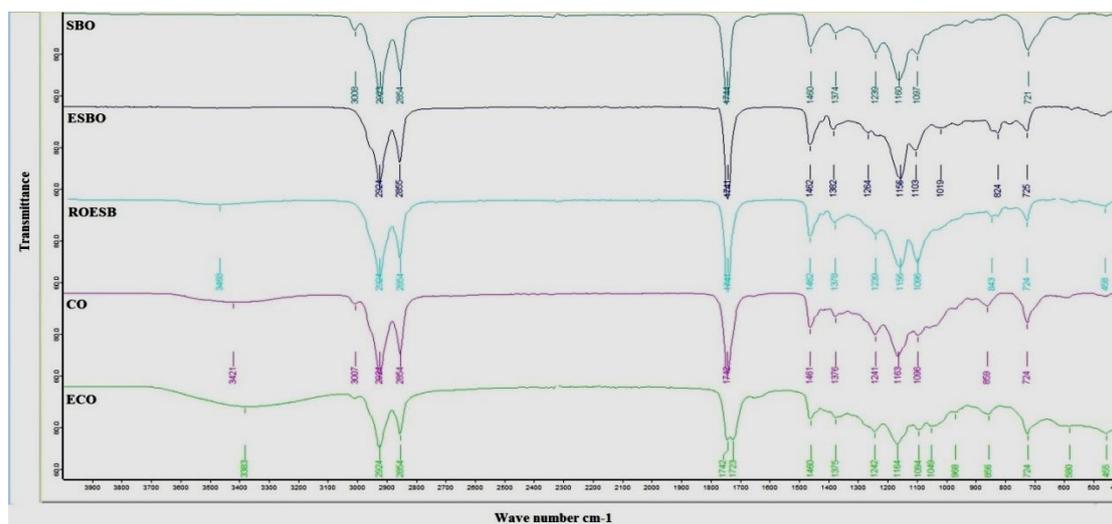
**Table 4: Characteristic features of polymeric dispersants**

	Solubility in solvents				Solid content	Water content
	Ethanol	Ethyl acetate	Toluene	Isopropanol		
PD1	S	S	S	S	98.6%	0.12%
PD2	S	S	S	S	96.5%	0.20%
PD3	S	S	S	S	99.1%	0.16%
PD4	S	S	S	S	99.8%	0.11%

S: Soluble

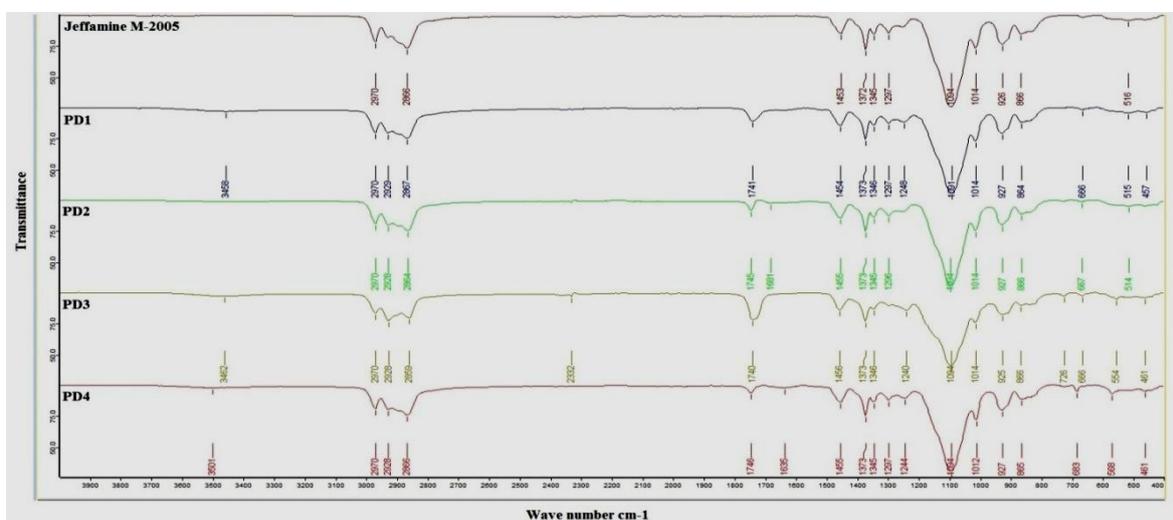
### 3.4. Fourier Transform Infrared (FT-IR) analysis

The FT-IR spectra in **Figure 5** show the disappearance of double bond group peak at  $3009\text{ cm}^{-1}$  demonstrated the conversion of double bond of oils to the epoxy group. Formation of epoxy group in the spectrum of fingerprint region was indicated by the peaks at  $843$  and  $824\text{ cm}^{-1}$ . The C=O ester peak of the triglyceride appears at  $1743\text{ cm}^{-1}$  and the hydroxyl peak at  $3460\text{ cm}^{-1}$ .



**Figure 5: The FTIR spectra of soybean oil (SBO), castor oil (CO), epoxidized soybean oil (ESBO), partially ring opened epoxidized soybean oil (ROESBO) and epoxidized castor oil (ECO).**

The FTIR spectra of polymeric dispersants in **Figure 6** show characteristic bands of Jeffamine® M2005: at  $2875\text{ cm}^{-1}$  and  $2970\text{ cm}^{-1}$  (for the stretching vibrations of  $\text{CH}_2$  and  $\text{CH}_3$  groups, respectively), at  $1450\text{ cm}^{-1}$  and  $1370\text{ cm}^{-1}$  (for the bending vibrations of  $\text{CH}_2$  and  $\text{CH}_3$  groups, respectively), at  $1090\text{ cm}^{-1}$  (for the ether C–O–C band of Jeffamine® M2005), at  $1743\text{ cm}^{-1}$  (for C=O ester peak of the triglyceride) and  $3458\text{ cm}^{-1}$  (for the hydroxyl group, OH).



**Figure 6: The FTIR spectra of polymeric dispersant PD1, PD2, PD3, PD4 and Jeffamine M-2005.**

### 3.2.3 Molecular weight

Number average molecular weight (Mn) and molecular weight polydispersity index (PDI) were affected by the ring opening of oxirane groups. **Table 5** represents the GPC results.

**Table 5: The gel permeation chromatography (GPC) data of polymeric dispersant**

Sample	Mn	Mw	PDI
STD	2.3867e <sup>3</sup>	4.9785e <sup>3</sup>	2.08
Soybean oil	1.2673e <sup>3</sup>	1.3394e <sup>3</sup>	1.05
Castor oil	1.3698e <sup>3</sup>	1.4543e <sup>3</sup>	1.06
Jeffamine	2.5189e <sup>3</sup>	3.1357e <sup>3</sup>	1.24
PPG2000	2.6125 e <sup>3</sup>	2.9186 e <sup>3</sup>	1.11
PD1	4.1034e <sup>3</sup>	5.1535e <sup>3</sup>	1.25
PD2	4.2816e <sup>3</sup>	5.4831e <sup>3</sup>	1.28
PD3	3.7386e <sup>3</sup>	5.1733e <sup>3</sup>	1.38
PD4	3.2449e <sup>3</sup>	3.7023e <sup>3</sup>	1.14

From **Table 5**, the result of GPC refers that at least one molecule of jeffamine react with one epoxide group.

### 3.3 Printed film properties

#### 3.3.1 Printing ink formulation, application

The printing ink was formulated according to the formulation given in **Table 1**. All ink formulations were milled on shaker after addition of glass beads, and then they were applied on polypropylene film using hand coater.

The properties of printing inks related to standard ink with the prepared polymeric dispersant are summarized in **Table 6** before and after the stability test.

**Table 6: Properties of printing ink**

PD	Mechanical		Optical properties							
	Curling	Adhesion	Transparency%		ΔS %		ΔE %		Gloss %	
			B*	A*	B*	A*	B*	A*	B*	A*
STD	No Curling	A	Used as a Standard for X-rite, Gloss meter							
PD1	No Curling	A	98.3	86.4	94.1	91.9	1.12	2.5	<b>96.7</b>	<b>96.5</b>
PD2	No Curling	A	99	97.8	94	92.1	2	2.8	<b>100</b>	<b>95.8</b>
PD3	No Curling	B	98.3	96.4	92.78	83.8	2.5	3.8	<b>91</b>	<b>87.8</b>
PD4	No Curling	A	109.6	96.8	92.29	88.1	1.2	1.9	<b>100</b>	<b>98.2</b>

A: no ink removal; B: less than 5% ink removal; C: more than 5% ink removal; D: failed.

A\*: After stability; B\*: Before stability.

The data reported in **Table 6** indicated that polymeric dispersants were prepared successfully by the partial substitution of petroleum polymers with vegetables oil derivative. Castor oil and soybean oil gave very comparable mechanical and optical properties.

#### 4. Conclusions

The present study demonstrated, the preparation of polymeric dispersants based on castor oil and soybean oil via epoxidation of oils followed by ring opening with different ring opening agents such as methanol, polypropylene glycol, and polyether amine. The prepared polymer was used in ink formulation to evaluate its efficiency. The results obtained were evaluated compared with commercial polymeric dispersant. It indicates that all prepared dispersants show high optical, mechanical properties and high stability for ink before and after stability test.

The polymeric dispersant 2 (PD2) and polymeric dispersant 4 (PD4) gave the best results (optical and mechanical) among the prepared polymer.

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### ملخص البحث باللغة العربية

تحضير وتوصيف بوليمرات مشتته معتمدة علي الزيوت النباتية لتطبيق أحبار الطباعة  
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يتضمن البحث تحضير بعض البوليمرات التي تستخدم كمشتتات للمخضبات والتي تعتمد في تحضيرها علي الزيوت النباتية مثل زيت الصويا وزيت الخروع حيث تتميز هذه الزيوت بتوافرها ورخص ثمنها. يتم تحضير البوليمر عن طريق عملية الأكسدة للروابط الثنائية بجزيئات الزيت ثم اضافة البوليمر ليقوم بفتح الحلقة الثلاثية ومثال علي ذلك استخدام البولي بروبيلين جليكول. ثم استخدام البوليمرات التي تم تحضيرها في تركيبات للاحبار كمشتت للمخضبات و تقييم أدائها من حيث الخواص اللونية والخواص الميكانيكية للحبر. من نتائج البحث يتضح ان كل البوليمرات التي تم تحضيرها لها أداء جيد جدا مقارنة بالبوليمرات المستخدمة صناعيا من حيث الخواص اللونية والخواص الميكانيكية.

