



Preparation and Evaluation of Polyamides for Printing Ink

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CrossMark

NON-reactive polyamides were prepared by reacting Soy-based dimer fatty acids and, two different diamines (ethylenediamine and isophoronedimine). Soy-based dimer fatty acids used was of dimer/trimer/ Monomer mixtures, $\geq 80/ \leq 18/ \leq 5$. The number average molecular weight of polyamide (Mn) ranged from 2479-2803, weight average molecular weight (Mwt) from 6761-10220, Z weight average molecular weight (Mz) from 13532-26876, maximum peak (Mp) from 5555-7448 and the most fundamental characteristic of a polymer its molecular weight distribution(PDI) degree of polymerization ranged from 2.727-3.713, the melting point (Tm) 106.477, the glass transition (Tg) was 76.46-98.9°C, the viscosity of 35% solution in butanol / toluene 1:1, v/v, 85-128 s at 25°C (Ford Cup No. 4) , the Softing point was 114-123°C. and Evaluation gloss and color strength polyamides in ink on films (paper, polyethylene milk, and polyvinylchloride).

Most of these polyamides have properties comparable to the commercially available polyamides used in flexographic inks.

Keywords: Polyamides, Dimer-fatty acid, Aliphatic and Cycloaliphatic diamines.

Introduction

The number of efforts in order to modify the properties of polyamide in recent times because of increasing interest of polyamide in high-tech applications, Polyamides with amide groups – NH-CO- in the main chain are considered to be one of the most important super-engineering materials because of their superior mechanical properties at elevated temperature due to their thermal stability[1].

Polyamides may be synthesized either by (A) polycondensation of divalent carboxylic acid and divalent amines, or by (B) polycondensation of difunctional amino acids containing both one amine and one carboxylic acid functionality in the same molecule (or their intramolecular ring-shaped condensation products “lactams”)[2].

Aromatic polyamides have received considerable attention with regard to the production of high performance materials due to their outstanding thermal stability, chemical resistance and electrical and mechanical properties [3-5].

The aliphatic polyamides are produced on a much larger scale than the fully aromatic polyamides and are the most important class of engineering thermoplastics [6]. The polymer fibers, nylon (nylon 6 and nylon 6.6) fibers are widely used in carpet factories [7-8].

Semi-aromatic polyamides consist of both aliphatic and aromatic fragments in the polymer main chain. It has excellent dimensional stability, low creep at elevated temperatures and good chemical resistance [9].

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Soy-based dimer acids have been traditionally used to synthesize and formulate hot-melt adhesives, flexographic inks, functional coatings, and engineering materials. Because of their wide applications, the investigations of soy-based polyamides are growing. There are many advantages of using soy-based dimer acids as starting materials, compared with using other monomers because they are nontoxic, less expensive, and potentially biodegradable. On the other hand, despite extensive research of formulations using different aliphatic amines, the condensation polymerization of a dimer acid with cycloaliphatic amines has not been extensively studied [10].

Fatty polyamides can be broadly classified into two categories: non-reactive and reactive. The non-reactive polyamides have a far lesser number of primary and secondary amine groups to react with epoxy resins compared to the reactive ones. These polyamides also differ in their properties and therefore in their end uses. Non-reactive polyamides are mostly solids and are used in the printing ink industry, mainly for flexographic and rotogravure inks, where hardness and solvent release is required. They are also used in over-print varnishes, adhesives and heat-seal coatings. Reactive polyamides are liquid in nature and are used in the surface coatings and adhesives industry [11,12].

Dimerisation of unsaturated fatty acids is an important process in the oleochemical industry. In this liquid phase batch process not only dimers of fatty acids are formed, but also trimers and isomers of the monomers. All product groups have a number of applications, with for dimer acids, the most important one being as components in polyamide [13].

1,2-Diaminoethane, commonly known as ethylenediamine (EDA), is a synthetic colorless to yellowish liquid at normal temperature and pressure. It is strongly alkaline and is miscible with water and alcohol. The main use for EDA is as an intermediate in the manufacture of tetraacetylenediamine, ethylenediaminetetraacetic acid (EDTA), organic flocculants, urea resins, and fatty bisamides. It is also used, to a much smaller extent, in the production of formulations for use in the printed circuit board and metal finishing industries, as an accelerator/curing agent in epoxy coatings/resins [14].

Isophoronediamine [IPDA] can be used in all typical amine reactions, such as reaction with carboxylic acid, phosgene, aldehyde, ketones and epoxides. It is miscible in all proportions at room temperature with a wide range of compounds such as water, alcohol, ester, ether, ketone as well as many aliphatic, aromatic and halogenated hydrocarbon. IPDA is diamine with special structure due to multiple alkyl substituted cyclohexane ring, amino group with different reactivity and cis-trans configuration. When compared to other commercially available amines, different properties become apparent in the properties of its derivatives and polymer compounds [15].

There are a large number of patents [16-20] on the preparation of polyamides with varied properties.

Inking polyamide studies underlined the effect of EDA and IPDA on the films which improved the gloss and color strength.

DAPAE (2) represent on Dimer fatty acid with ethylenediamine, (Number of carbon in the diamine block) and DAPAIPDA (10) represent on Dimer fatty acid with isophoronediamine.

The present work, (a) the preparation of two polyamide for use in the flexographic ink industry with dimer acids and ethylenediamine; or dimer acids and isophoronediamine (b) measurement of the number average molecular weight (M_n), weight average molecular weight (M_w), Z Higher average molecular weights (M_z), maximum peak (M_p), degree of polymerization (DP), the glass transition temperature, the melting point temperature, the end group titration method; and (c) evaluation in flexographic printing ink the flexographic inks presently in use are applied to Paper, Polyethylene milky and polyvinylchloride films.

Experimental

Materials

Dimer acid supplied from Yantai Sunny Chem International Co, Ltd (China) ($\geq 80/\leq 18/\leq 5$) dimer/trimer/monomer acids respectively.

Ethylenediamine and Isophoronediamine, 99% pure, laboratory reagent grade, supplied from Aldrich Chemical Company (USA).

Alcohol soluble polyamide Standard supplied from colorchem company (China).

Perchloric acid 0.1N, alcoholic potassium

hydroxide, Bromophenol blue, methyl orange, n-Butanol, toluene, xylene, chloroform, anhydrous isopropyl alcohol and ethanol supplied from Sigma-Aldrich - Germany

Methods

Polyamide resins were prepared with two different types diamines (Ethylenediamine, Isophoronediamine)

Polyamide synthesis from Dimer fatty acid and Ethylenediamine

A reaction mixture consisting of equivalent weight of dimer fatty acids ($\geq 80/\leq 18/\leq 5$ dimer/trimer/monomer acids respectively) to diamine was charge in metallic reactor and equipped with a thermowell, mechanical stirrer, dropping funnel, and nitrogen inlet. The mixture was stirred and heated from (120-140 °C). At this temperature aqueous ethylenediamine, equivalent to the amount of dimer acid was added dropwise over a period of 45 to 90 min. After completion of amine addition, the mixture was heated gradually to 200 °C. An amber-colored, brittle solid resin

was obtained. The scheme of preparation is given in Fig.1. [12].

Polyamide from dimer fatty acid and isophoronediamine

A reaction mixture consisting of equivalent weight of dimer fatty acids ($\geq 80/\leq 18/\leq 5$ dimer/trimer/monomer acids respectively)) with charge in metallic reactor and equipped with a thermowell, mechanical stirrer, dropping funnel, and nitrogen inlet. The mixture was stirred and heated from (120-140 °C). At this temperature aqueous isophoronediamine, equivalent to the amount of dimer was added dropwise over a period of 45 to 90 min. After completion of amine addition, the mixture was heated gradually to 260°C in 3 h and kept at this temperature for another 5 h under vigorous stirring. An amber-colored, brittle solid resin was obtained

There is no chemical formula in the library

Characterization

The Fourier transfer infrared (FTIR) spectra were obtained using a Perkin Elmer spectrum 100FT-IR spectrophotometer.

TABLE 1. Effect of different diamine of reactant Effect of change in mole ratio of reactants.

Serial number	Dimer fatty acids dimer/trimer/monmer acids	Diamine	Ratio acid/amine equavelent	Acid value	Amine value	glass transition (°C)	Mwt	Viscosity (s)	softing point (°C)
1	$\geq 80/\leq 18/\leq 5$	Ethylene diamine	1:1	8	5.3	98.9	6761	85	114
2	$\geq 80/\leq 18/\leq 5$	Isophornediamie	1:1	9	7.4	93.57	10220	128	123

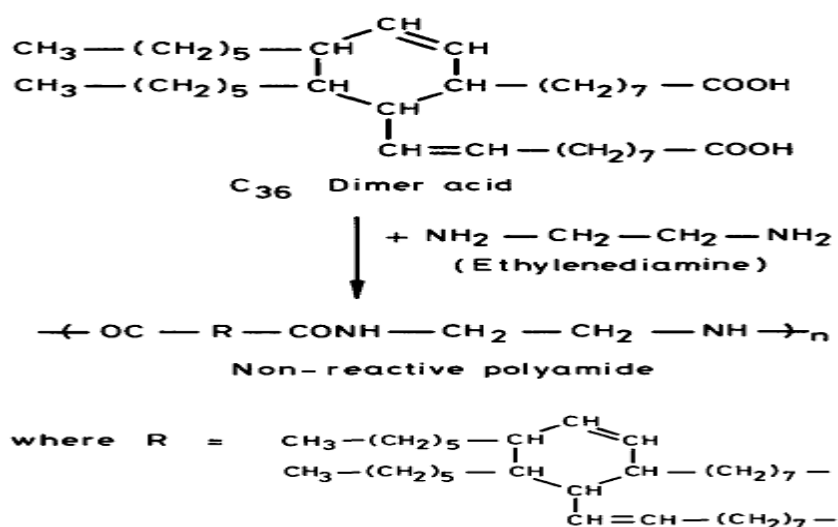


Fig. 1. Scheme for the preparation of non-reactive polyamide.

Different scanning calorimetric (DSC) was determined on (Setram DSC Evo 131) under a nitrogen flow at a heating rate of 5 °C / min).

Gel permeation chromatography (GPC) was determined on pump 515 waters refractive index detector 2410 waters columns: styragel columns.

Determination of acid value according to ASTM D 974 -12. In addition, Determination of amine value was measured through ASTM D2074-07-13. On the other hand, viscosity of prepared inks was investigated by ASTM D1200 Ford No 4, Determination of softening point ASTM E 2858. Where Dry film gloss measurement ASTM D 523.

The gloss degree was measured by a gloss meter of type Minigloss10N model of Sheen Instruments company from United Kingdom, with a measuring geometry 60 Degrees, resolution 0.1 gloss unit and accuracy ± 1.0 gloss unit (against reference standard). Moreover, Spectrodensitometer ASTM D 730508.

The color strength was measured on a Spectrodensitometer of type X-rite Nixpro model of X-rite Company from United States, with an measuring geometry 45°/0°, Spot Size at Sample 3.4mm (13in) standard and Measurement Range 0.00D–50D; 0–160%R with Measurement Time Approx. 1.4 seconds, approx.9 seconds for consecutive measurements in Speed Read mode and Repeatability ± 0.005

Results and Discussion

The current work is designed to be divided to four main parts, which could be summarized in the following points; i) the preparation of DAPAE (2); ii) preparation of and DAPAipda (10) iii) analyses by Laboratory equipments of the prepared polyamide; and finely, iv) evaluating the gloss degree and color strength after inking

FT-IR characterization

FT-IR spectra of the polyamides are shown in Fig. 2-4 the Standard beaks of NH stretching appears at 3299 cm^{-1} , where A symmetric and symmetric C-H (alkane) stretching appears at 2925.48 and 2854.13 cm^{-1} , amide II band, C-O stretching appears at 1641.13 cm^{-1} , CH₂ symmetric deformation appears at 1458 cm^{-1} , CH₂ Asymmetric deformation appears at 1374.03 cm^{-1} and -CH=CH-(cis-), (CH₂)_n-bending appears at 589.147 cm^{-1} .

The DAPAE (2) beaks of NH stretching appears at 3434.6 cm^{-1} , where A symmetric and symmetric C-H (alkane) stretching appears at 2925.48-2856.0 cm^{-1} , amide II band, C-O stretching appears at 1631.48 cm^{-1} , CH₂ symmetric deformation appears at 1450 cm^{-1} , CH₂ Asymmetric deformation appears at 1386.57 cm^{-1} and -CH=CH-(cis-), (CH₂)_n-bending appears at 576.612 cm^{-1} .

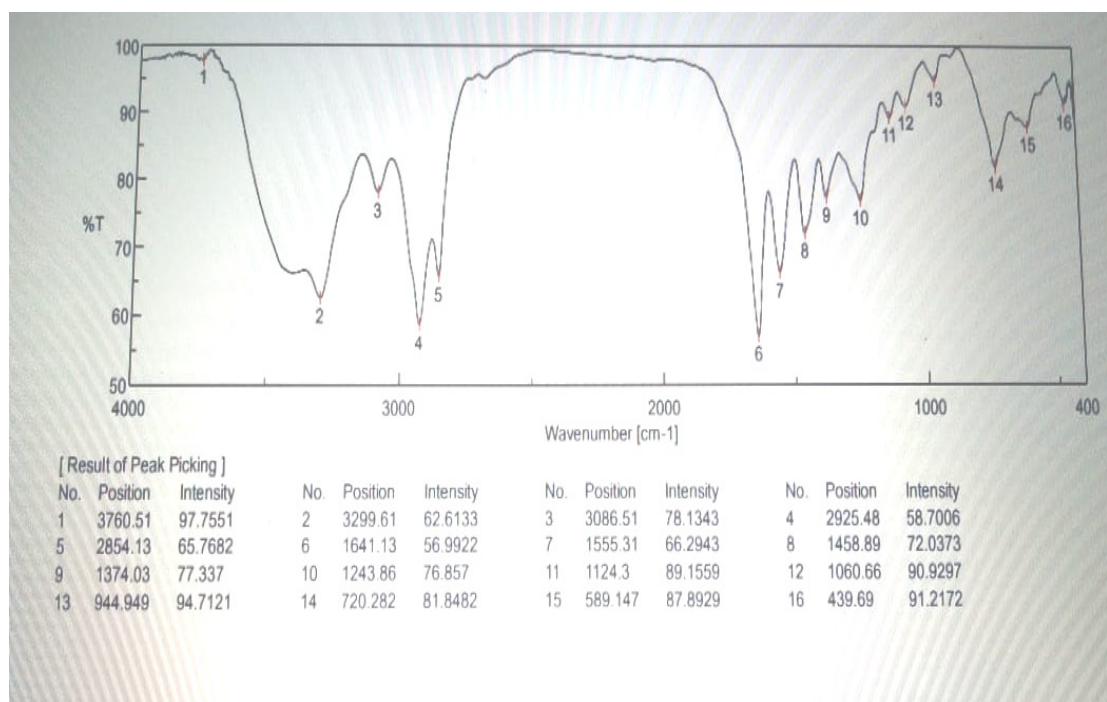


Fig. 2. FT-IR –Spectral data represented for standard polyamide.

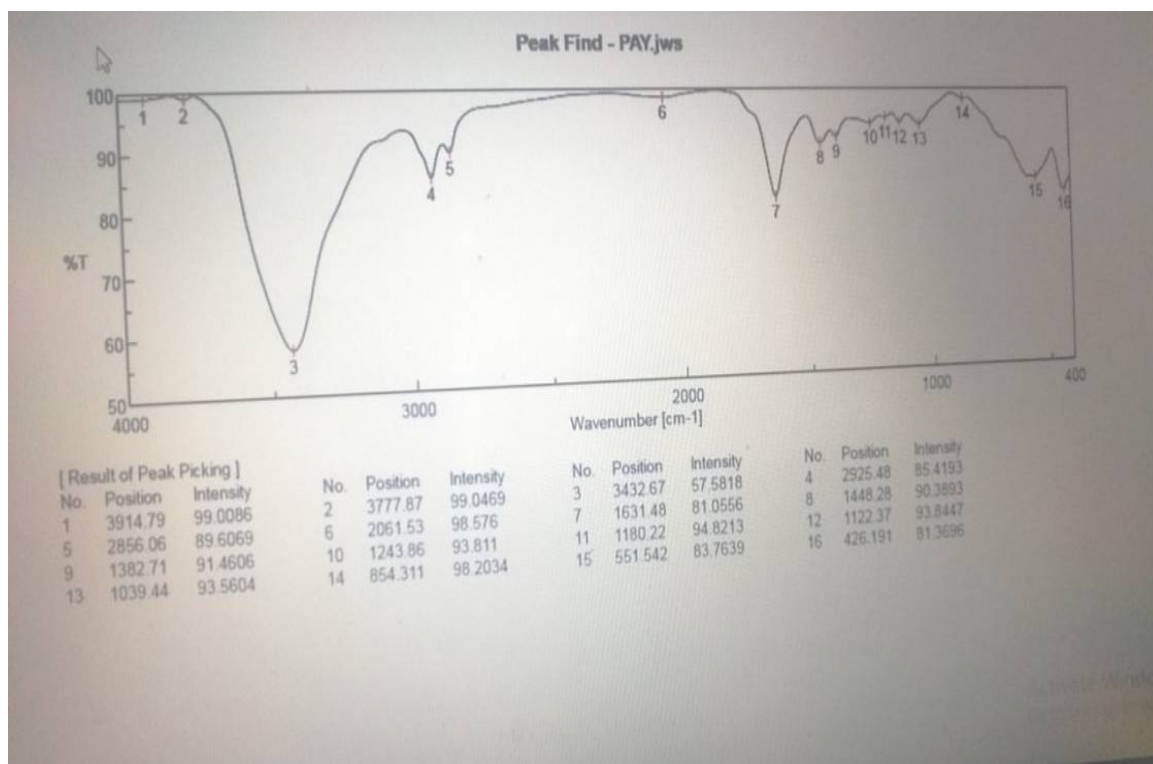


Fig. 3. FT-IR –Spectral data represented for DAPAE(2)

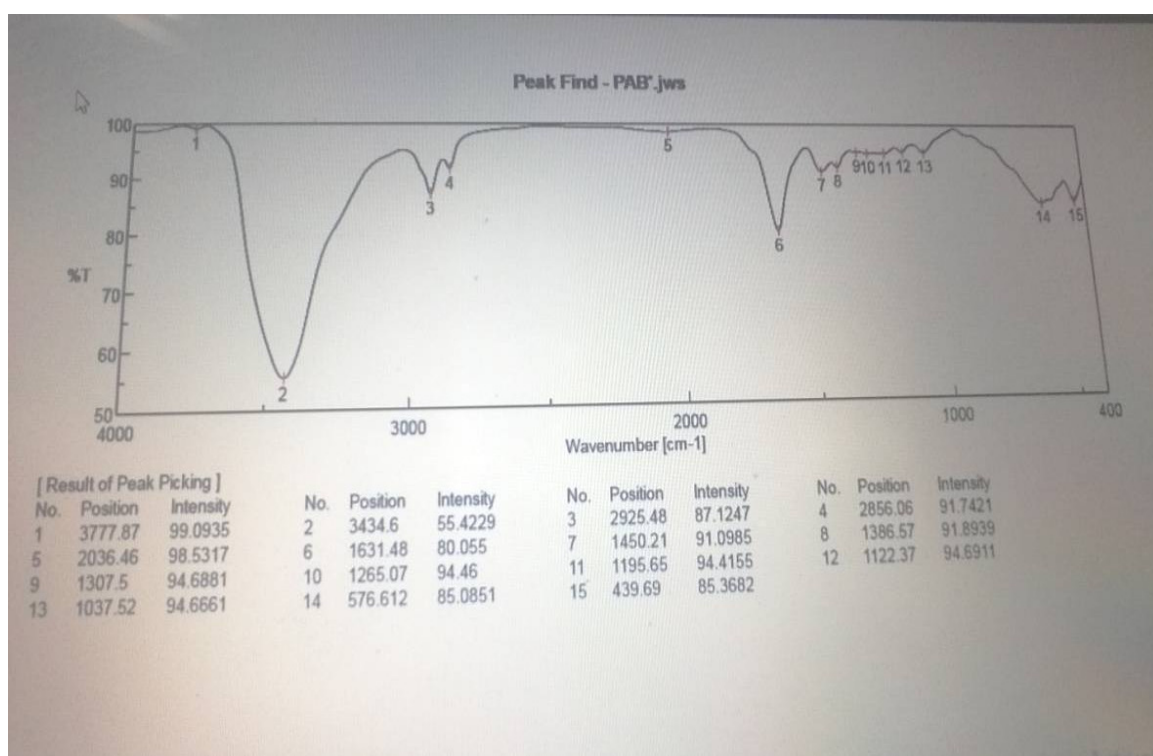


Fig. 4. FT-IR –Spectral data represented for DAPAipda (10).

The DAPAipda (10) beaks of NH stretching appears at 3432.67 cm^{-1} , where A symmetric and symmetric C-H (alkane) stretching appears at 2925.48 and 2856.06 cm^{-1} , amide II band, C-O stretching appears at 1631.48 cm^{-1} , CH₂ symmetric deformation appears at 1448.28 cm^{-1} , CH₂ Asymmetric deformation appears at 1382.71 cm^{-1} and -CH=CH-(cis-), (CH₂) n-bending appears at 551.612 cm^{-1} [21].

Different scanning calorimetric

Different scanning calorimetric (DSC) is a technique used to investigate the response of the amorphous polymers. DSC can be used to study the melting of a crystalline polymer or the glass transition. The thermal stability of the polymer composites is the most important parameter for their processing and applications, so it is very instructive to characterize the thermal degradation behavior. DSC measures the difference in heat flow rate ($\text{mWt} = \text{mJ/sec}$) between a sample and inert reference as a function of time and temperature [22].

The DSC curves of the three samples are represented in Fig 5-7 and related thermal properties are given in Table 4. First, it can be seen that the Tg of Standard polyamide is

76.46C , and Tm is 119.75 , for the Tg of DAPAE(2) (ethylenediamine-based polyamide) is 98.942C and Tm is 106.477 , and the Tg of DAPAipda(10) (isophornediamine-based polyamide) is 93.574C . where the Tg represents the chain segments' motion in amorphous phase, as isophornediamine is more flexible than ethylenediamine, a higher Tg value for DAPAE(2) indicates that, the flexibility of segments in the aliphatic diamine monomer still plays an important role in effecting their Tg. [23]. Tg decreases as the number of carbon atoms the n-alkyl series increases. So the TgDAPAipda(10) is less than DAPAipda(10) [24-25].

Determination of molecular weight

Average molecular weight of polyamide from 4000-7000 [26]. The number average molecular weight (Mn), weight average molecular weight (Mwt), maximum peak molecular weight (MP), Z weight average molecular weight (Mz), and the most fundamental characteristic of a polymer its molecular weight distribution (PDI). The obtained results were shown in Table 5 and Fig. 8-10. The present molecular weight of dimer acid-based polyamides affected by reaction time and temperature.

TABLE 4. The glass transition temperature values of polyamide standard and the prepared polyamides.

	ST	DAPAE (2)	DAPAipda (10)
Tg	76.46	98.942	93.574
Tm	119.75	106.477	

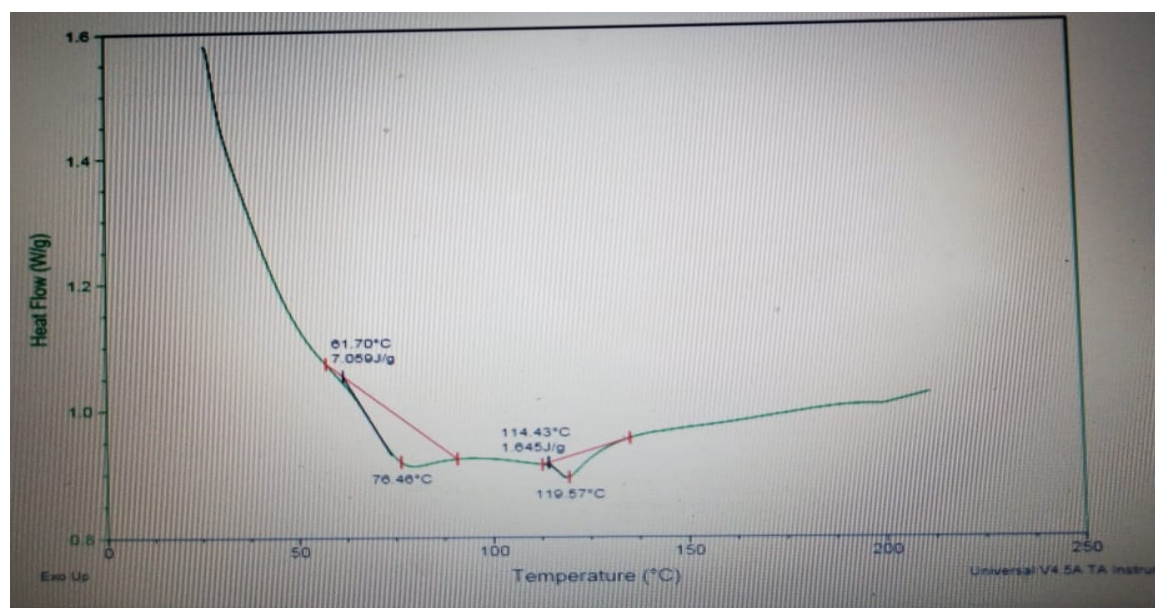


Fig. 5. Glass transition temperature for standard.

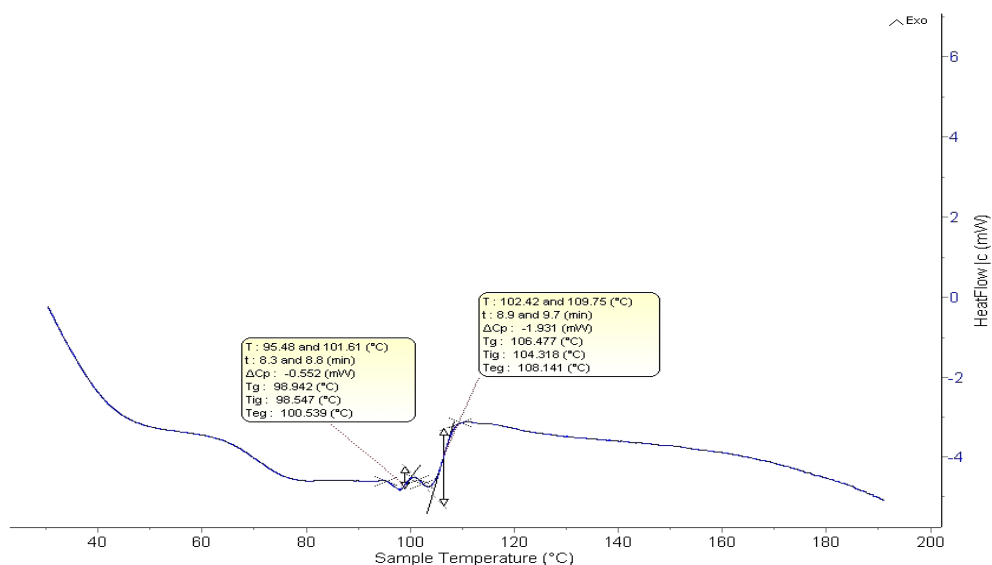


Fig. 6. Glass transition temperature for DAPAc(2).

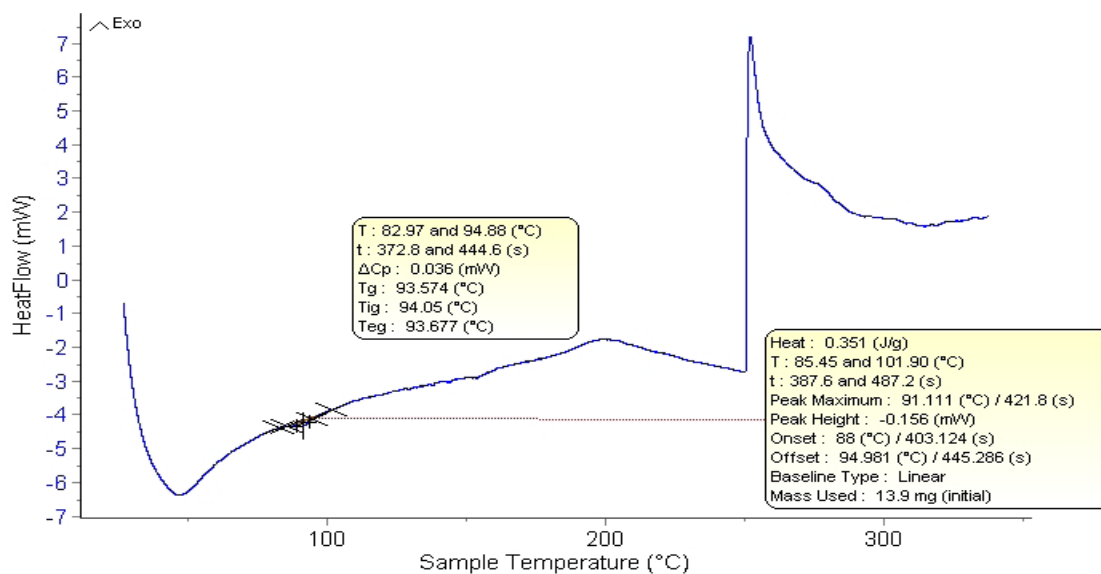
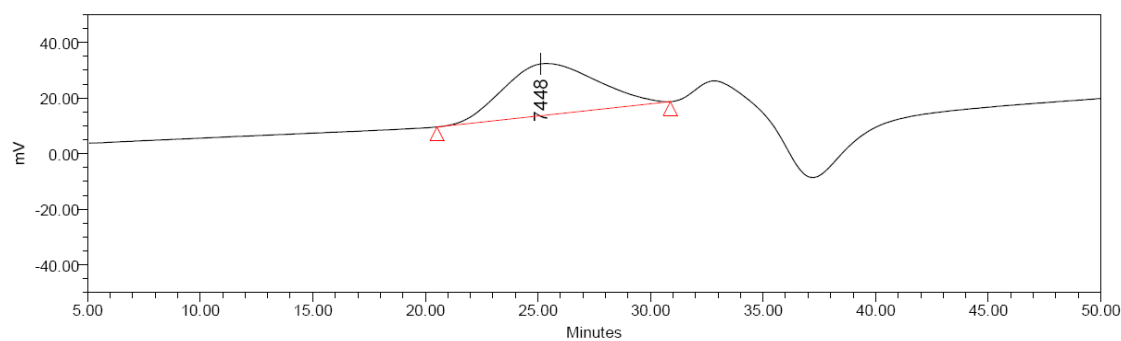


Fig. 7. Glass transition temperature for DAPAIpda (10).

TABLE 5. Show different Mwt Standard, DAPAc (2) and DAP Aipda (10).

Polyamide	Retention Time	Mn	Mwt	MP	Mz	Mz+1	PDI
ST	25133	2803	9590	7448	22447	36990	3.421
DAPAc (2)	25600	2479	6761	5555	13532	20579	2.727
DAP Aipda (10)	25167	2752	10220	7294	26876	47906	3.713



GPC Sample Results

	Retention Time	Mn	Mw	MP	Mz (Daltons)	Mz+1 (Daltons)	Poly-dispersity
1	25.133	2803	9590	7448	22447	36990	3.421

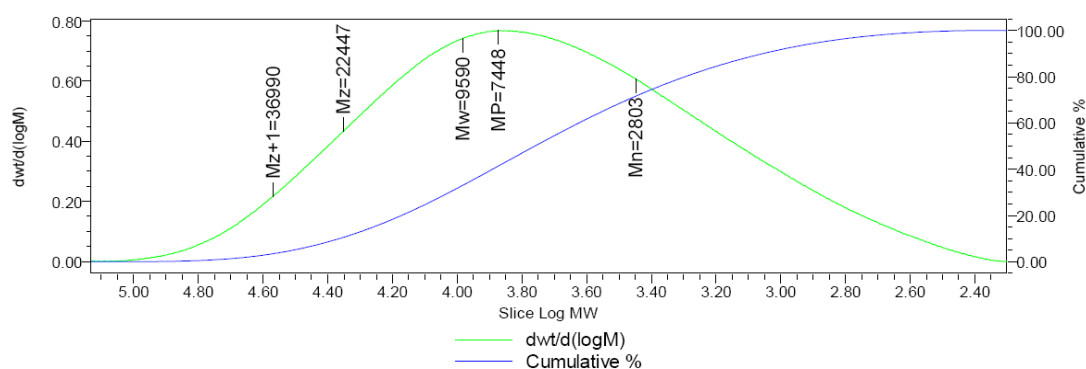
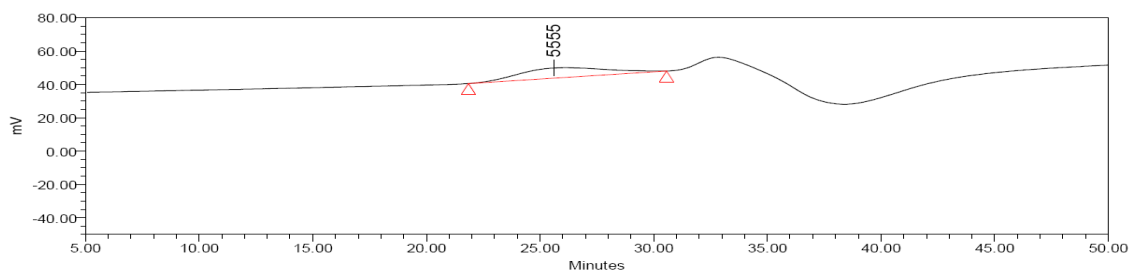


Fig. 8. Gel permeation chromatography for PA standard.



GPC Sample Results

	Retention Time	Mn	Mw	MP	Mz (Daltons)	Mz+1 (Daltons)	Poly-dispersity
1	25.600	2479	6761	5555	13532	20579	2.727

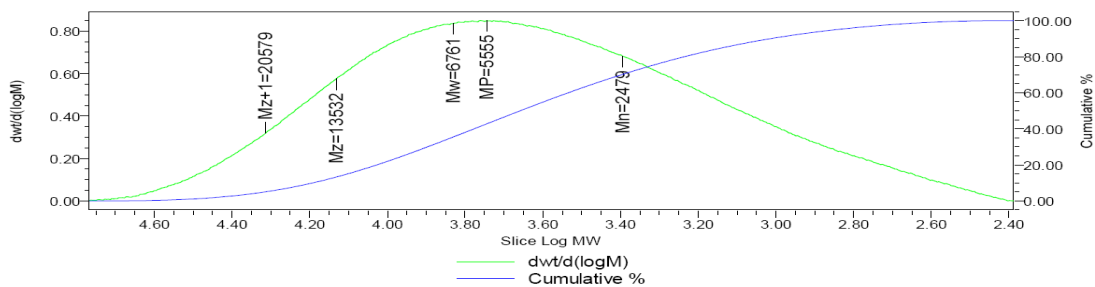


Fig. 9. Gel permeation chromatography for DAPAc (2).

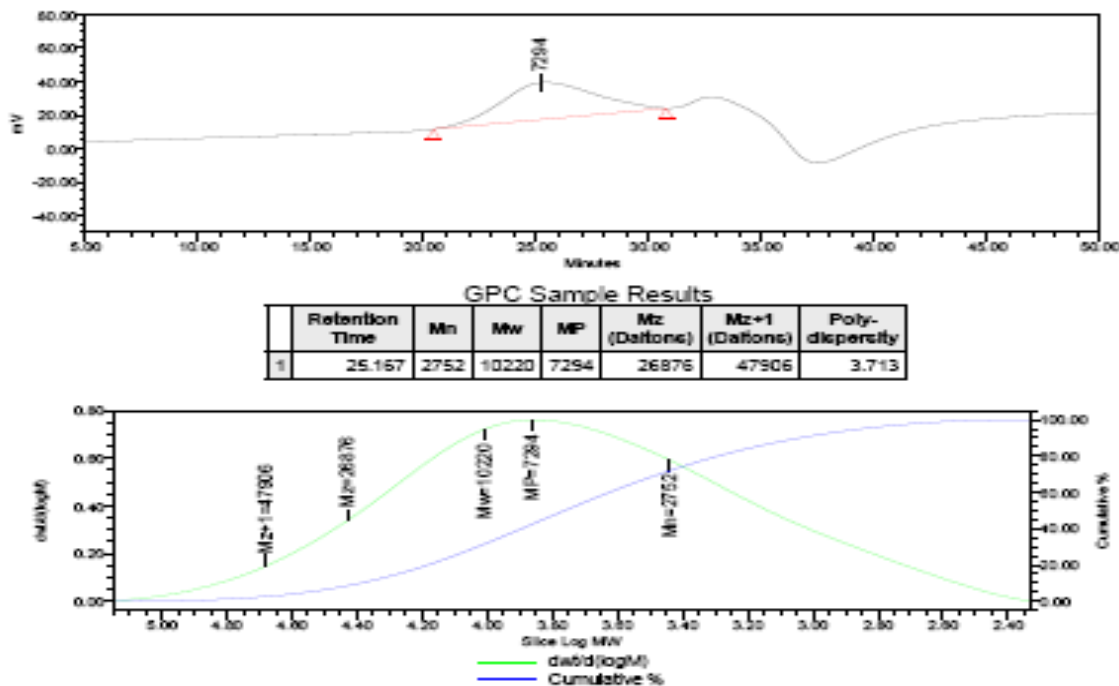


Fig. 10. Gel permeation chromatography for DAPAipda (10).

Evaluation of the synthesized of polyamide as resin in printing ink

The synthesized polyamides were applied in flexo printing inks formulations. The printing inks formula contains pigment, solvent and additives to achieve the desired properties such as, gloss and color strength. All synthesized polyamides were used in formulations in Table 6, 7 with using diverse organic pigments such Lithol Rubin to evaluate its efficiency against standard. The printing process was applied with glass rods of 1mm diameter by using Red-Devil shaker for 60 minutes

Dry film gloss measurement

The Gloss is the surfaces that causes them to have shiny or lustrous, metallic or matte appearances. Gloss values of printed films are very important value to explain the performance of synthesized polyamide. Gloss of printed film with flexographic ink made by synthesized polyamides were evaluated and recorded in Table 8.

Flexographic ink on paper coat

The result of gloss meter test as showing in Figure 11, the gloss of polyamide standard was 43 , the gloss of DAPA (2) was 39 and the gloss of DAPA (6) was 42. It can be observed that polyamides of standard and DAPA (6) are better gloss than DAPA (2) .

Flexographic ink on polyethylene milky film

The result of gloss meter test as showing in Figure 12 .the gloss of polyamide standard was 43, the gloss of DAPA (2) was 52 and the gloss of DAPA (6) was 36.8.

Also it can observed that polyamides of Standard and DAPA(2) are better gloss than DAPA(6) and the gloss of DAPA(2) is better than standard .

Flexographic ink on polyvinylchloride film

The result of gloss meter test as showing in Figure 13. The gloss of polyamide standard was 49, the gloss of DAPA(2) was 46 and the gloss of DAPA(6) was 69. it can observed that polyamides of DAPA(6) is better gloss than Standard and DAPA(2), and the gloss of standard is better than DAPA(2)

Spectrodensitometer

Delta-E is a metric used to determine the visible difference or distance between two colors. It is very useful for sorting “closeness” of paints to a scanned sample. The Delta E76 refers to a formulas that was first standardized in the year 1976. now using a more accurate formula that takes into account new and improved models of the human eye; this formula is Delta E 2000. Even though the Delta E 76 formula is still very popular, Nix Sensor Ltd. recommends always using the Delta E 2000 formula for maximum accuracy and excellent real world results.

TABLE 6. Prepare polyamide varnish.

Material	%
Polyamide alcohol grade	40
Ethanol	30
Isopropanol	20
Isobutanol	5
Toluene	5
Total	100

TABLE 7. Prepared polyamide varnish in ink.

Material	Standard	DAPA(2)	DAPA(6)
Lithol Rubin (Pigment)	15	15	15
Polyamide resin (40%solid content)	60	60	60
ATBC	5	5	5
Ethyl acetate	20	20	20
Total	100	100	100

TABLE 8 . Gloss of printed ink with different films by using synthesized polyamides compared to standard.

No.	Polyamide	paper Coat	Polyethylene milk	Polyvinylchloride
1	Standard	43	43	49
2	DAPA(2)	39	52	46
3	DAPA(6)	42	36.8	69

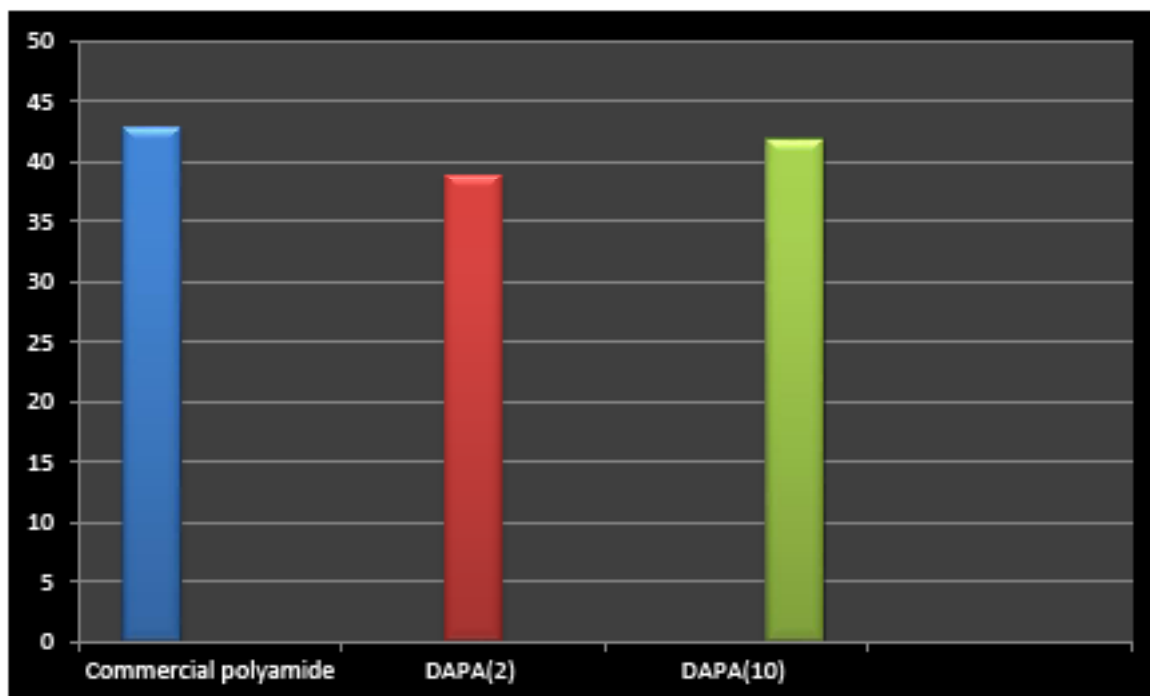


Fig. 11. Gloss of Lithol Rubin flexographic ink prepared with synthesized polyamides on paper.

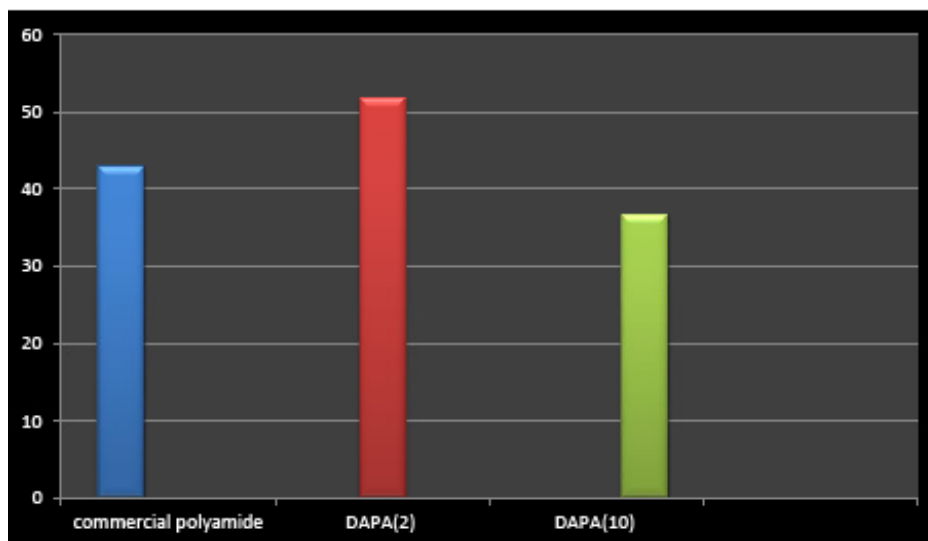


Fig. 12. Gloss of Lithol Rubin flexographic ink prepared by synthesized polyamides in polyethylene milky.

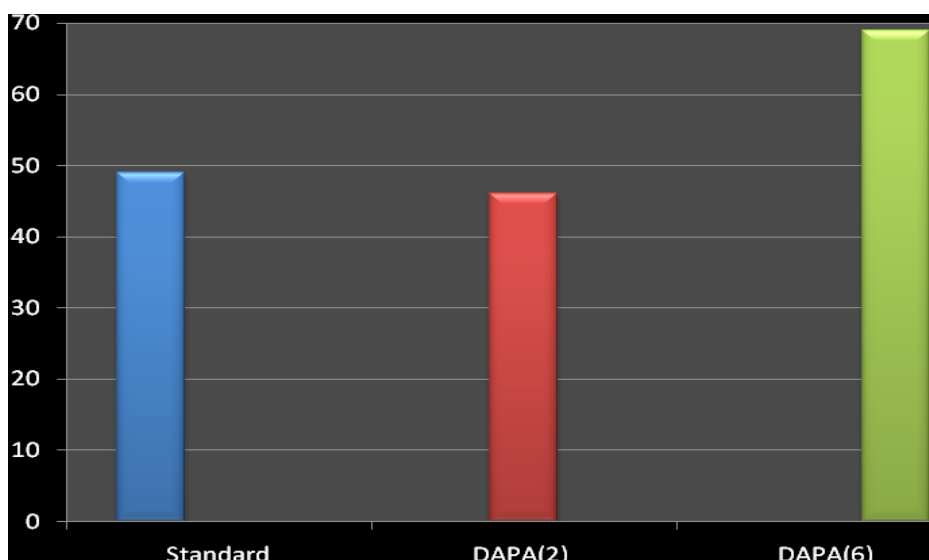


Fig. 13. Gloss of Lithol Rubin flexographic ink prepared by synthesized polyamides in polyvinylchloride film.

The result as shown in Table 9 and Fig. From 14 -21.

If Delta Ae2000 equal 2.5 the result approve

If Delta Ae2000 less than 2.5 the result more approve

If Delta Ae2000 higher than 2.5 the result less approve

Color strength flexographic ink on paper coat

Color strength of DAP Ae (2) against Standard was more approve, where Delta Ae2000 was 1.75, Color strength of DAP Aipda (10) against

Standard was less approve, where Delta Ae2000 was 4.32

Color strength flexographic ink on polyethylenemilky film

Color strength of DAP Ae (2) against Standard is more approve, where Delta Ae2000 was 2.27m Color strength of DAPAipda (10) against Standard was more approve, where Delta Ae2000 was 2.49

Flexographic ink on polyvinylchloride film

Color strength of DAPAE(2) against Standard is the less approve, where Delta Ae2000 was 7, Color strength of DAPAipda(10) against Standard was approve, where Delta Ae2000 was 1.8

The most result approve except (St/(DAPAE(2))) was less approve on pvc film and St/DAPAipda(10) on paper

Conclusion

Non-reactive polyamides have been prepared from two different of diamines(ethylenediamine or from isophornedimine) and dimer fatty acid. In these products the number average molecular

weight (Mn) ranged was 2479-2803, weight average molecular weight (Mwt) was 6761-10220, Z weight average molecular weight(Mz) from 13532-26876,maximum peak (Mp) from 5555-7448, degree of polymerization ranged was 2.727-3.713, the viscosity of 35% solution in butanol / toluene 1:1,v/v, 85-128 s at 25°C, the softing point was 114-123°C. Most of these polyamides have properties comparable to commercial polyamides (acid and amine value <10 mg KOHg-1), which find use in flexographic inks. The glass transition temperature was 76.46-98.9°C. Both of DAPAE(2) and DAPAipda(10) were interpretation of physical properties, FTIR. Evaluation gloss and color strength polyamide

TABLE 9. Show the results of spectrodensitometer on different films.

Film	Comparison	Delta Ae 2000	Deltae 76	Figure
Paper	St/DAPAE (2)	1.75	4.30	14
Paper	St/DAPAipda (10)	4.32	8.99	15
PE milk	St/(DAPAE (2))	2.27	7	16
PE milk	St/DAPAipda (10)	2.49	6.98	17
PVC	St/(DAPAE (2))	7	14	18
PVC	St/DAPAipda (10)	1.8	2.3	19

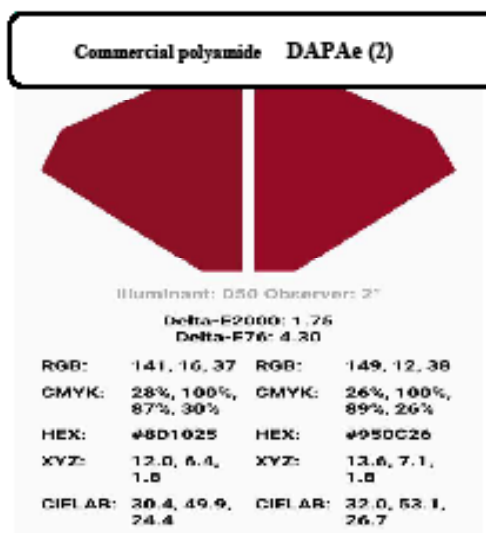


Fig. 14. Comparison between St/(DAPAE(2)) ... paper substrate

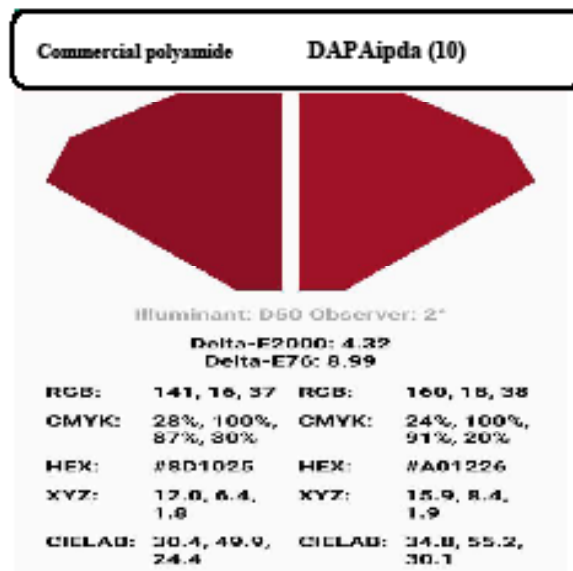


Fig. 15. Comparison between St/DAPAipda(10) in paper substrate.

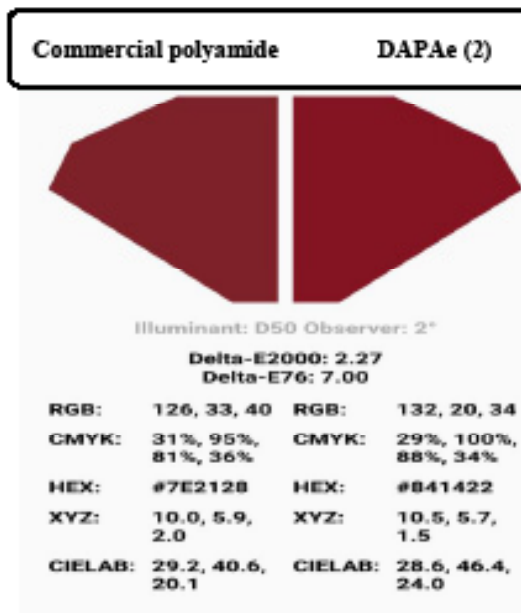


Fig. 16. Comparison between St/DAPAE(2) in polyethylene milky substrate .

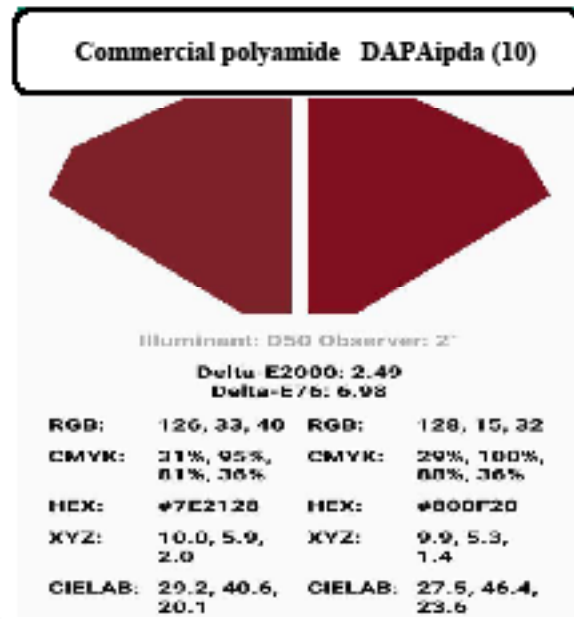


Fig. 17. Comparison between St/DAPAIPDA(10) in polyethylene milky substrate .

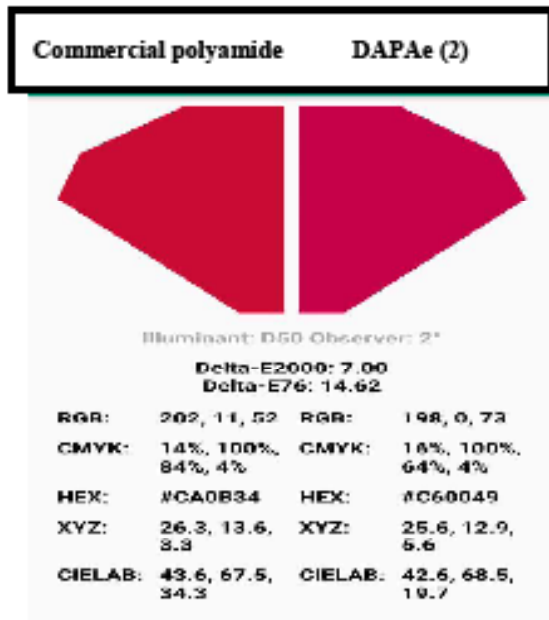


Fig. 18. Comparison between St/DAPAE(2) in polyvinylchloride substrate .

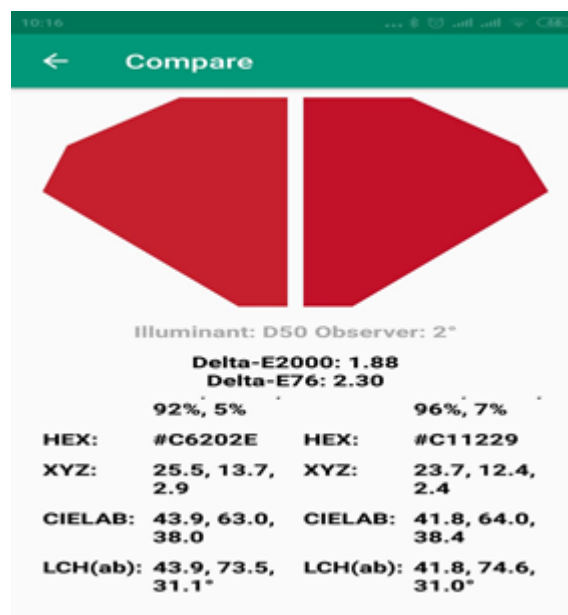


Fig. 19. Comparison between St/DAPAIPDA(10) in polyvinylchloride substrate .

in ink comparison DAPAE(2), DAPAipda(10) and ST in films (paper, polyethylene milk, and polyvinylchloride). the gloss of DAPA(2) is better than standard on polyethylene film, the gloss of DAPAipda(10) is better than standard on pvc film. Color strength of DAPAE(2) against Standard is more approve, and Color strength of DAPAipda(10) against

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تحضير وتوصيف البولى اميد الكحولى المستخدم فى حبر الطباعة

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نلاحظ فى الأونة الأخيرة زيادة الجهود المبذولة من أجل تعديل خصائص البولى اميدو ذلك من خلال استخدام ثنائى الحمض الدهنى وثنائى الامين من أجل زيادة التطبيقات التكنولوجية المتطورة للبولى اميد. تهدف هذه الدراسة التحضير نوعين من البولى اميد باستخدام ثنائى الحمض الدهنى المحضر من زيت الصويا وتفاعل ثنائى الحمض الدهنى مع نوعين من ثنائى الامين (الايثيلين ثنائى الامين والايروفورون ثنائى الامين). و قد تمت دراسة تأثير ثنائى الامين على الخواص وذلك بتوصيف البولى اميد ب استخداما لتحليل الحرارى والوزن الجزيئى والتحليل الطيفى بالأشعة تحت الحمراء والرقم الحامضى والرقم الامينى. كما تمت دراسة تأثير ثنائى الامين على احبار البولى اميد. أظهرت الدراسة تحسين للمعه على افلام البولى الايثيلين باستخدام الايثيلين ثنائى الامين وتحسين للمعه على افلام البولى فينيل كلوريد باستخدام الايزوفورون ثنائى الامين وقوة لون احبار البولى اميد باستخدام الايثيلين ثنائى الامين على سطح الورق والبولى ايثيلين وكذلك قوة اللون لاحبار البولى اميد باستخدام الايزوفورون ثنائى الامين على سطح البولى فينيل كلوريد كما أظهرت عملية التوصيف مدى زيادة درجة التليين للبولى اميد وتقارب الرقم الحامضى والرقم الامينى.