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CHEMICAL SCIENCES

The effect of linseed oil/canola oil blend on the coating and thermal properties of waste PET-based alkyd resins

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Abstract: This study aims to prepare oil-modified alkyd resins using a linseed oil/canola oil (LO/CO) blend and waste PET depolymerization product, suitable for environmentally friendly coating applications. Waste PET flakes obtained from grinding post-consumer water bottles were depolymerized by the aminoglycolysis reaction at high pressure. Raw depolymerization product (DP) was used in the synthesis of four components, 50% oil alkyd resins by monoglyceride method. DP has partly replaced the dibasic acid component in the PET-based alkyd formulations. Besides PET-based alkyds, reference alkyds without DP were also synthesized for comparison. Then, the surface coating properties and thermal behaviors of alkyd films were determined. The effect of DP usage and the changing ratios of LO/CO blend on coating properties and thermal behaviors of alkyd films were investigated. In addition, the optimum LO/CO blend ratio which is compatible with alkyd formulation was attempted to be determined. At the end of this study, glossy, soft/medium-hard films were obtained with excellent adhesion, impact strength, and chemical resistance. Thermal resistance and final thermal oxidative degradation temperature increased with adding DP to the alkyd formulation. Using LO/CO blend in the formulations affected oxidation rate and ratio, hence, drying time/degree and oxidative stability of alkyd films.

Key words: Poly(ethylene terephthalate), depolymerization, alkyd resin, linseed oil, canola oil, coating.

INTRODUCTION

Alkyd resins, which constitute the most crucial class of binders, are one of the main components of the paint and coating industry. Alkyd resins are polyesters obtained from the condensation of oils or fatty acids, polyacids, and polyalcohols (Ploeger et al. 2008). Alkyd resins are frequently preferred in both solvent-based and water-based paint systems with their superior properties (Hofland 2012, Jones 2003, Hlaing & Oo 2008). The chemical structure of the oil used in alkyd resin synthesis is very important. The type and amount of oil or fatty acid, in other words, the number of unsaturated groups in its structure significantly affect the surface coating properties of the alkyd film (Bender 2013). The physical and chemical surface coating properties of alkyd resins can be significantly improved with various modifications. Alkyds can be modified with various chemicals or polymeric materials, affecting their final film properties. In the technical literature, there are many studies on epoxy, vinyl, acrylate, urethane, styrene, phenolic, and silicon-modified alkyds synthesized by various modification reactions (Ploeger et al. 2008, Hofland 2012, Jones 2003, Akgün et al. 2016). Unlike the modifications made with such different chemicals, there are studies in the literature on modifying the oils or fatty acids, which are the main components of the alkyd resin, and synthesizing the alkyd resins using modified oils or fatty acids (Ogunniyi & Odetoye 2008, Aigbodion & Okieimen 2001, Mukhtar et al. 2007, Ikhuoria et al. 2004, Akintayo & Adebowale 2004, Issam & Cheun 2009, Kumar et al. 2010, Boruah et al. 2012, Odetoye et al. 2012, Patel et al. 2008, Bora et al. 2014, Okieimen & Aigbodion 1997, Ezeh et al. 2012, Nimbalkar & Athawale 2010, Bajpai & Seth 2000, Issam et al. 2011, Ikhuoria et al. 2011). In these studies; alkyd resins with various oil percentages were synthesized using modified oils, regional oils, or acrylated oils. On the other hand, alkyd resins containing waste PET intermediates are also available. There are various methods for the evaluation of PET wastes. These are; adding wastes to primary material at a certain rate during production, mixing or blending wastes with other polymers, producing raw materials from wastes via different chemical reactions in the presence of various reactants, and the conversation of heat generated by incineration of wastes into various types of energy (Paszun & Spychaj 1997, Ghosal & Nayak 2022, Karayannidis & Achilias 2007). Some of the chemical recycling processes applied for the production of chemical raw materials from PET wastes are hydrolysis (Karayannidis et al. 2002, Güçlü et al. 2003a), glycolysis (Güçlü et al. 1998), hydrolysis-glycolysis (Güçlü et al. 2003b), aminoglycolysis (Acar & Orbay 2011, Acar et al. 2013a, b) and aminolysis (Awodi et al. 1987, Shukla & Harad 2006, Spychaj et al. 2001, Achilias et al. 2011, Tawfik & Eskander 2010, Bulak & Acar 2014). Monomeric or oligomeric intermediates obtained from these depolymerization reactions of PET have been used in the syntheses of the solvent based alkyd (Bulak & Acar 2014, Ertaş & Güçlü 2005, Güçlü & Orbay 2009, Tuna et al. 2013, Khan & Chandra 1995, Spychaj 2002, Atta et al. 2013), water based alkyd (Acar et al. 2013b, Güçlü 2010), alkyd-amino (Torlakoğlu & Güçlü 2009), polyester polyol (Vaidya & Nadkarni 1988, Sabnis et al. 2012), polyurethane (Acar & Orbay 2011, Patel et al. 2005, Shamsi et al. 2009), unsaturated polyester (Vaidya & Nadkarni 1987, Aslan et al. 1997, Farahat et al. 2000, Suh et al. 2000, Öztürk & Güçlü 2004), epoxy (Bal et al. 2017) and epoxy ester (Çam et al. 2015).

In this study, waste PET and linseed oil (LO)/canola oil (CO) blend-based alkyd resins were synthesized successfully for environmentally friendly coating applications. To the best of our knowledge, an experimental study has not been reported yet on the synthesis of LO/CO blend modified alkyd resin, incorporating waste PET aminoglycolysis product as the dibasic acid component of four-component alkyd resin in the literature. In this respect, our study differs from other similar studies. In the synthesis of waste PET and LO/CO blend-based alkyd resins, the use of the aminoglycolysis product of waste PET partially instead of the dibasic acid component was carried out for the first time in this study. In addition, the optimum LO/CO blend ratio was determined in this working range for good coating performance and thermal resistance.

MATERIALS AND METHODS

Materials and instruments

In the depolymerization reactions, waste PET flakes (the sieve fraction is 8-10 mesh) obtained from grinding post-consumer PET water bottles were used. The chemicals used in the aminoglycolysis reactions, triethylamine (TEA) and 1,4 butanediol (1,4 BDO) were Merck synthesis grade. The xylene and zinc acetate used in the depolymerization reactions were synthesis or analytical grade. The other chemicals (pyridine, potassium hydroxide, acetic anhydride, sodium hydroxide, hydrochloric acid, and isopropyl alcohol) which were used for various analyses, were also Merck synthesis or analytical grade. Linseed oil (LO) was supplied from Serkim (Turkey). Refined and winterized canola oil (CO) (Aro brand cooking oil) manufactured by Yonca Food Industry (Turkey) was purchased from the market. The other chemicals used for alkyd resins synthesis reactions, phthalic anhydride (PA) and glycerine (GLY), were also obtained from Merck (Germany). In addition, the dryers (zirconium naphthenate, 6% and cobalt naphthenate, 6%) were kindly provided by AKPA Kimya (Turkey). The rest of the materials were the synthesis grade or analytical grade. Distilled water was used where necessary throughout the study.

Depolymerization reactions were realized using the High-Pressure Reactor (Autoclave, Berghoff, BR-1000, Germany). Thermal analysis of the depolymerization product was determined using Differential Scanning Calorimetry (DSC, SII Exstar 6000/DSC 6200, Japan). Thermal behaviors of the alkyd resins were investigated using the Thermogravimetric Analyzer (TGA, Linseis STA PT 1750, TGA/DTA combined device, Germany). The surface coatings properties of the resin films were tested by the Drying Time Tester (Erichsen, 415/E model, Germany), Pendulum Hardness Tester (Sheen, König model, UK), Cross-Cut Adhesion Tester (Erichsen, GS 10 model, Germany), Falling Sand Abrasion Tester (Erichsen, 2511-11 model, Germany), Impact Tester (BYK Gardner, PF-1115 Light-Duty model, Germany) and Glossmeter (Sheen, 101 N mini model, UK).

Depolymerization of waste PET and characterizations of depolymerization product (DP)

Depolymerization of waste PET flakes (8-10 mesh) was carried out by simultaneous aminolysis-glycolysis (aminoglycolysis) reaction. In aminoglycolysis reaction, PET/1,4 BDO/TEA molar ratio was 0.5/1/1, and depolymerization reaction was realized at high temperature (235-240°C) and high pressure in the presence of xylene (200 mL) for 3 h. Zinc acetate (ZnAc) (1% by weight of PET) was used as a catalyst. At the end of the reaction time, the raw depolymerization product (DP) was taken from the autoclave and the xylene phase was removed by decantation. No further purification was done for DP. Then, DP was dried in a vacuum oven at 40-60°C. The conditions of depolymerization reaction of waste PET are summarized in Table I.

For the characterization of DP; acid value (AV), hydroxyl value (HV), and amine value (AMV) was determined by volumetric methods according to ASTM D-1639, ASTM E-222 and ASTM D-2074, respectively. The findings about AV, HV and AMV of raw depolymerization product (DP) of waste PET are presented in Table II.

In addition, DSC analysis of DP was also performed. DSC measurement was carried out in a nitrogen atmosphere, with ~10 mg sample, by heating from room temperature to 300°C, at a rate of 10°C/min. DSC curve of depolymerization product (DP) of waste PET is presented in Figure 1.

As seen in Figure 1, there are broad melting peak and small peaks/shoulders in the DSC curve of DP sample. The observation of multiple broad and small peaks and/or shoulders rather

Table I. The condition of depolymerization reaction of waste PET.

Depolymerization reaction type	PET/1,4 BDO/TEA molar ratio	Temperature (°C)	Pressure (Bar)	Time (h)	Symbol of raw product
Aminoglycolysis	0.5/1/1	235-240	13-17	3	DP

than a single sharp peak can be attributed to different oligomeric structures with different end groups.

The possible reactions realized during the simultaneous aminolysis-glycolysis reaction of waste PET, using triethylamine (TEA) and 1,4 butanediol (1,4 BDO), are presented in Reaction 1 and Reaction 2.

Various side reactions may also occur during the depolymerization reaction. The etherification side reactions occur by yielding oligomers with water and hydroxyl-end groups or oligomers with hydroxyl and carboxylic acid end groups. Another important side reaction is the hydrolysis reaction, which takes place by the water formed in the other side reactions or by the water present as an impurity. In addition, the terephthalamide, which is formed at the end of the aminolysis reaction, reacts with water, and products containing carboxyl end groups are obtained. Thus, as a result of all these main and side reactions, oligomers are probable occur having hydroxyl-hydroxyl, carboxyl-carboxyl, hydroxyl-carboxyl, amine-hydroxyl, amine-carboxyl and amine-amine end groups (Acar & Orbay 2011, Acar et al. 2013a).

DSC curve of DP also support this situation. More than one endotherm (broad melting peaks, small shoulders or peaks) is seen in the DSC curve of DP. This indicates that the resulting product is a mixture of oligomers rather than a pure product. In order to predict the oligomers in the composition of DP; these values obtained from the DSC curve can compared with the melting temperatures of PET oligomers given in the literature (Brandrup & Immergut 1996) and it can be determined which oligomer the endotherms observed in the DSC curves might belong to. When the DSC curve of DP presented in Figure 1 was examined, a small peak at 55°C, an interfering broad endotherm extending between 70 and 220°C, and a broad peak centered at

Table II. The functional group content of raw depolymerization product (DP) of waste PET.

Raw depolymerization product	Acid value (AV) (mg KOH/g)	Hydroxyl value (HV) (mg KOH/g)	Amine value (AMI) (mg KOH/g)
DP	112	204	69

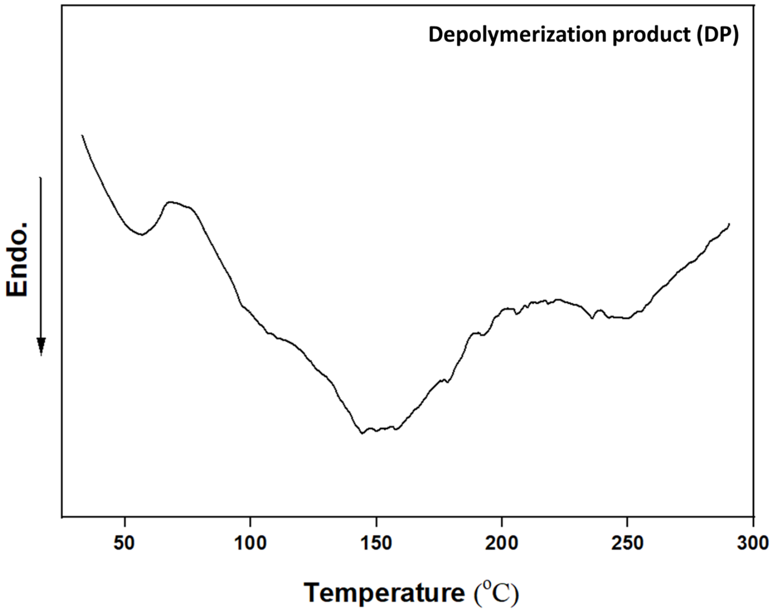


Figure 1. DSC curve of depolymerization product (DP) of waste PET.

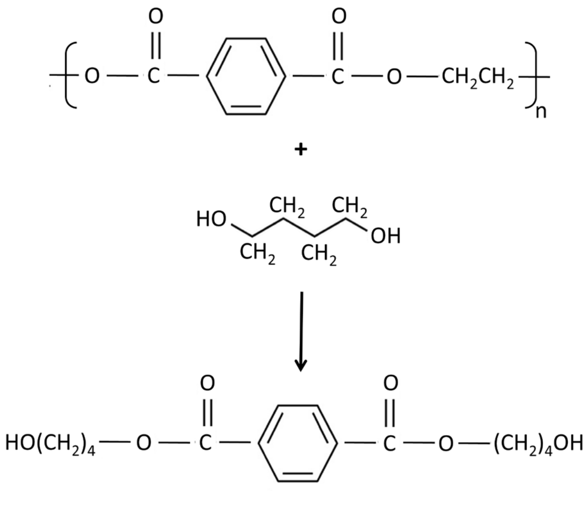
250°C between 230 and 270°C were observed. In this context, the composition/content of this raw depolymerization product consisting of the monomer/dimer/oligomer mixtures can be determined by comparing the melting points of the DP sample with that of the PET oligomers taking into account the shifting of these peaks and their interference with each other (Acar et al. 2013a, Kasap-Yegen et al. 2023).

As a result, this depolymerization product is an oligomer mixture, and it contain hydroxyl, carboxyl, and amine functional groups. In this study, this hydroxyl and carboxyl functional aminoglycolysis product was used instead of the dibasic acid component in the alkyd resin synthesis according to acid equivalent. In the calculations, the hydroxyl group content was deducted from the base equivalent in the formulation.

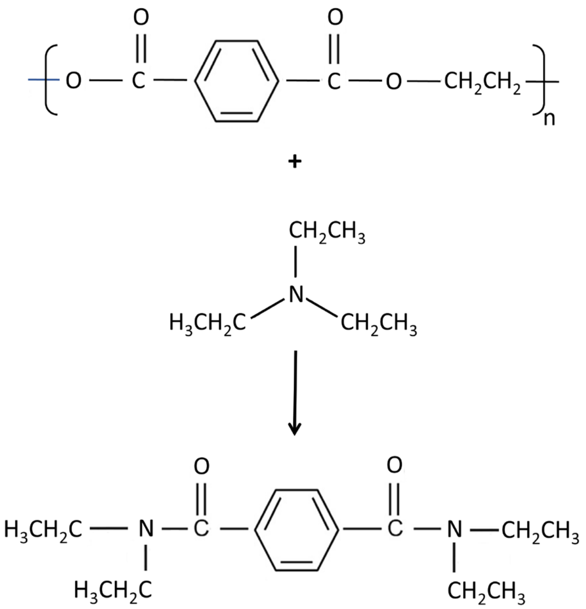
Formulation calculations of alkyd resins

The “K alkyd constant system” was used for the formulation calculations of the alkyd resins according to the literature (Patton 1962). The components and symbols of the synthesized alkyd resins are presented in Table III.

Four-component PET-based alkyd resins formulated to have an oil content of 50% were prepared using different ratios of the LO/CO blend. In these formulations, DP was used in a 10% by equivalent wt. instead of PA. That is, a partial replacement was achieved using DP instead of the dibasic acid component. Since DP contains both hydroxyl and carboxyl groups it has both acid equivalent (e_a) and base equivalent (e_b). Therefore, in the formulation calculations, to maintain the acid-base balance, the base equivalent of the DP was deducted from the base equivalent of diol component (1,4 BDO). In addition, reference alkyd resins without DP were synthesized in the same reaction conditions of PET-based alkyds for comparison.



Reaction 1: The main expected reaction of PET in the case of glycolysis with 1,4 BDO.



Reaction 2: The expected reaction when using TEA in the aminolysis of PET.

Table III. The components and symbols of all synthesized oil-modified alkyd resins.

Symbols	Alkyd Resin Components			
	Monobasic acid*	**Dibasic acid***	**Diol**	**Triol**
Reference alkyds				
REF-Alk-100	%100 LO	PA	1,4 BDO	GLY
REF-Alk-90	%90 LO +%10 CO	PA	1,4 BDO	GLY
REF-Alk-80	%80 LO +%20 CO	PA	1,4 BDO	GLY
PET-based alkyds				
PET-Alk-100	%100 LO	%90 PA + %10 DP	1, 4 BDO	GLY
PET-Alk-90	%90 LO +%10 CO	%90 PA + %10 DP	1, 4 BDO	GLY
PET-Alk-80	%80 LO +%20 CO	%90 PA + %10 DP	1, 4 BDO	GLY

LO: linseed oil, CO: canola oil, PA: phthalic anhydride, 1,4 BDO: 1,4 butanediol, GLY: glycerine, DP: depolymerization product of waste PET (AV: 112 mg KOH/g; HV:204 mg KOH/g), * % ratios are based on equivalent weight.

Table IV. Formulations of reference oil-modified alkyd resins.

	Equivalent weight (E)	Acid equivalent (e_a)	Base equivalent (e_b)	Amount (g)
REF-Alk-100				
LO (100%)	280	0,1450	-	41
PA	74	0,3201	-	24
GLY	31	-	0,4490	14
1,4 BDO	45	-	0,0859	4
REF-Alk-90				
LO (90%)	280	0,1305	-	37
CO (10%)	280	0,0145	-	4
PA	74	0,3201	-	24
GLY	31	-	0,4490	14
1,4 BDO	45	-	0,0859	4
REF-Alk-80				
LO (80%)	280	0,1160	-	33
CO (20%)	280	0,0290	-	8
PA	74	0,3201	-	24
GLY	31	-	0,4490	14
1,4 BDO	45	-	0,0859	4

The K constant was 1.07, and the R value (ratio of base equivalent to acid equivalent, $R=e_b/e_a$) was 1.15.

Details of the reference and PET-based alkyd resins formulations are presented in Table IV and Table V, respectively.

Synthesis reactions and acid values of alkyd resins

The two-stage alkyd resin synthesis reactions were carried out in the five-necked glass reactor equipped with a mechanical stirrer,

Table V. Formulations of PET-based oil-modified alkyd resins.

	Equivalent weight (E)	Acid equivalent (e_a)	Base equivalent (e_b)	Amount (g)
PET-Alk-100				
LO (100%)	280	0,1600	-	45
PA (90%)	74	0,2746	-	20
GLY	31	-	0,4102	13
1,4 BDO	45	-	0,0689	3
DP (10%)	501 (E_a), 274 (E_b)	0,0305	0,0558	16
PET-Alk-90				
LO (90%)	280	0,1440	-	40
CO (10%)	280	0,0160	-	5
PA (90%)	74	0,2746	-	20
GLY	31	-	0,4102	13
1,4 BDO	45	-	0,0689	3
DP (10%)	501 (E_a), 274 (E_b)	0,0305	0,0558	15
PET-Alk-80				
LO (80%)	280	0,1280	-	36
CO (20%)	280	0,0320	-	9
PA (90%)	74	0,2746	-	20
GLY	31	-	0,4102	13
1,4 BDO	45	-	0,0689	3
DP (10%)	501 (E_a), 274 (E_b)	0,0305	0,0558	15

Equivalent weights (E_a and E_b) of DP1 were calculated using AI and HV (AV: 112 mg KOH/g; HV:204 mg KOH/g).
The K constant was 1.1, and the R value (ratio of base equivalent to acid equivalent, $R=e_b/e_a$) was 1.15.

gas inlet, thermometer, reflux condenser + Dean-Stark part. In the first stage of synthesis (the alcoholysis reaction) partial esters were prepared from polyol with oil. For this, monobasic acid, diol, triol and catalyst (KOH in methanol, 0.1% by wt. of total charge) were loaded into the reactor together with xylene (8-10% by wt. of total charge). Then the temperature was gradually increased to around the 220-240°C range with continuous stirring (200-250 rpm) in the nitrogen atmosphere. Monoglyceride formation was monitored by the methanol test. Since the triglyceride, and diglyceride are relatively insoluble in methanol, when a methanol/sample volume ratio of 2 to 1 gives a clear solution, the completion of the alcoholysis reaction was accepted. For the second stage of synthesis (the esterification reaction) the remaining hydroxyl groups were reacted with dibasic acid. For this, the system was cooled to about 160-180°C, and the dibasic acid component was added to the reactor. Afterward, the temperature was raised to the 220-240°C range, and it was kept constant within this temperature range throughout the reaction. The progress of alkyd synthesis reactions was followed by acid value (AV) determination. For this purpose, samples were taken from the reactor at certain time intervals for AV analysis, and the reactions were continued until the acid value reached the desired value (~10 mg KOH/g).

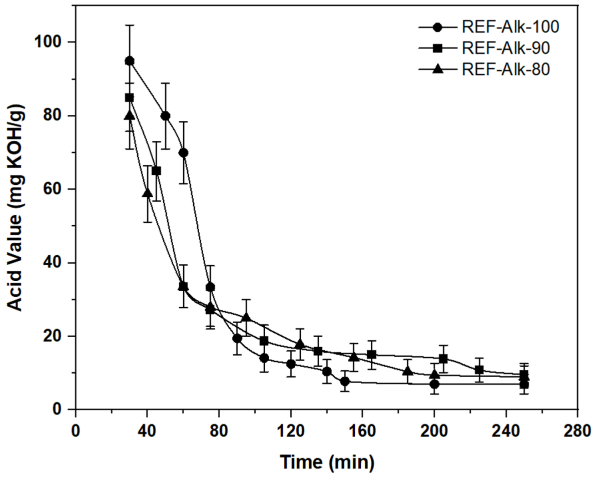


Figure 2. The change of acid value with reaction time for reference oil-modified alkyd resins.

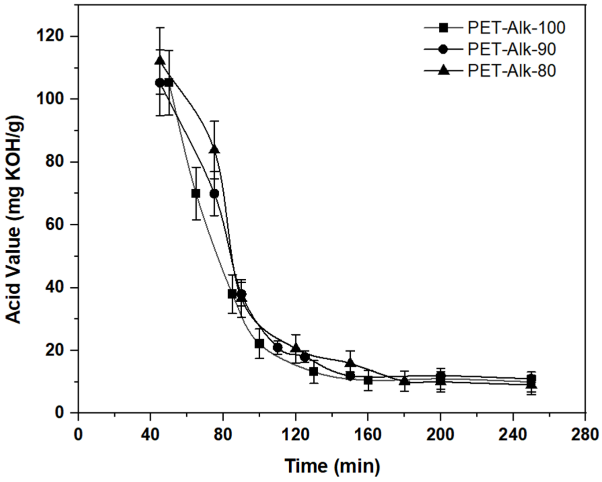


Figure 3. The change of acid value with reaction time for PET-based oil-modified alkyd resins.

The time-dependent acid value changes observed during the synthesis reactions of the reference and PET-based alkyd resins are presented in Figure 2 and Figure 3, respectively.

As seen in Figure 2 and 3, as the alkyd synthesize reactions progressed, the acid values decreased in time, and this decrease was more rapid during the early stage of the reaction than in the later stage. The different reactivities of the primary and secondary groups of the polyol explain these changes observed in acid values during polycondensation. Primary hydroxyl groups react faster than secondary hydroxyl groups of polyol (Goldsmith, 1498). Therefore, it was previously stated in the literature that the rapid decrease in acid values observed in the early stage of the reaction corresponds to the period in which the primary hydroxyl groups react, while the latter stage corresponds to the period when the secondary hydroxyl groups react (Aigbodion & Okieimen 2001, Ertaş & Güçlü 2005, Güçlü & Orbay 2009). In this study, a similar behavior was also observed for waste PET and linseed oil/canola oil blend based alkyd resins. As previously reported in the literature, the first part of the graph probably represents the formation time of the linear molecules, and the second part probably represents the formation of final alkyd structure (Aigbodion & Okieimen 2001, Okieimen & Aigbodion 1997).

Preparation of alkyd resin films

In order to perform the physical and chemical surface coating tests of the reference and the PET-based alkyd resins, in accordance with the relevant standards, resin films were prepared on different surfaces with different methods. For the water-resistance test, films were prepared by the casting technique on tin plates, and for the alkali resistance test, the immersion technique on wide glass tubes (diameter x height: 3x12 cm). For the impact resistance test, films were prepared on 10x15 cm (width x height) metal plates using the 50 μ applicator. For all remaining tests, films were prepared on 10x15 cm (width x height) glass plates using the 50 μ applicator. To prepare the resin films, firstly, all the resins were diluted to 70% with xylene. Then, the driers were added to the diluted alkyd resins in calculated amounts to contain 0.1% Co and 1% Zr by wt. of the total resin amount. Afterward, the resin films were oven-cured at 120°C for 1 hour. In addition, in order to observe the drying behavior of resin films in the air, another series of 50 µ films was also prepared on glass plates, and they were left under room conditions to dry in the air.

Determination of coating properties

Physical coating performance of all alkyd films were determined with the application of drying degree (drying time), hardness, adhesion strength, abrasion resistance, impact strength, and gloss tests. In addition, water resistance, acid resistance, alkali resistance, salt resistance, solvent resistance, and environmental resistance tests were also carried out in order to determine the chemical coating performance of the films. Details are presented below.

Physical surface coating properties

Drying degree test: (DIN 53150/ISO 9117-5). In this test method, which is applied according to Modified Bandow-Wolff Method, there are 7 drying degrees rated from 1 to 7, and it is determined whether coatings have reached these drying degrees and, if so, how long it took to reach them. Thus, this method allows the evaluation of both the drying (or dryness) degree and the drying speed. The drying stages/degrees are determined by adherence or non-adherence of beads or paper disks to the film under various loadings. The drying stages are as follows:

In *stage 1*, the small glass beads scattered over the coating can be easily and completely removed from the surface with a fine brush after 10 s. In *stage 2*, the paper disk placed onto the coating with a soft rubber disk does not adhere to the surface after loading 20 g for 60 s. For *stage 3*, a 200 g load is used for 60 s, and the paper disk does not adhere to the surface. For *stages 4 and 5*, a 2 kg load (corresponding to a plunger force of 20 N produced using lever pressure) is applied for 60 s to the coating surface. In stage 4, the paper disk does not adhere to the surface after loading, but a visible sign of paper is present on the surface, while in stage 5, any sign is not observed. *Stages 6 and 7* are also determined similarly, using a 20 kg load (corresponding to a plunger force of 200 N produced using lever pressure) during the 60 s.

In order to compare the drying test results obtained according to the Modified Bandow-Wolff test method with the Finger-Touch test method results (ASTM D 1640), the definitions of the drying terms in this method are summarized below in accordance with the literature. (Gardner & Sward 1972, Gooch 2011, Basic Coatings 2014, Kansai Altan 2023, Transocean Coatings 2023, Koleske 1995, Francis 2016, Harris 2005, Amstock 2000, Corrosionpedia 2023, HMG Paints 2023, AS/NZS standard 2002).

Dry-to-touch: When the film is lightly touched by a finger the coating does not adhere to the finger. *Surface-dry:* Only the skinned over the surface is dry and the underneath of the film is wet. When the film is pressed strongly with a finger, the coating moves/separates. *Dry:* The film is dry, however, the coating cannot be handled without damage. When the film is pressed strongly with a finger, fingerprints form on the coating. *Hard-dry:* The film is not displaced when the film-coated panel is pinched by a small and light force between the forefinger and thumb. *Through-dry:* The film is not displaced when the film-coated panel is compressed with medium force between the forefinger and thumb. *Full-dry:* The film is fully dry throughout its thickness. When strong force is applied to the film, the coating does not deteriorate or displace.

Hardness test (Pendulum damping test): (DIN 53157). In this test method, hardness is determined by measuring the mechanical damping time of the pendulum oscillating on the coating. Results are given in “König seconds” compared to 250 König seconds, which is the oscillation time (damping time) of the standard glass plate (the hardest material according for this standard) (Balci & Iyim 2014).

Adhesion strength test (Cross-hatch test): (ASTM D 3359-76). The purpose of this test is to determine how well the coating is bonding to the solid substrate. In this method, cuts at right angles to each other (6x6) are created on the coating surface with a 6-blade cross-cutter. Thus, a lattice pattern-shaped square is obtained on the coating surface. With the help of tape or brush, the coating separated from the surface is removed, and this pattern is compared with the schematic representations in the standard visually. Results are given as “adhesion%”.

Abrasion resistance test (Falling-sand test): (ASTM D 968-05). In this test method, silica sand (as abrasive) is dropped down onto the panel mounted at a 45^o angle through a vertical guide tube until the substrate becomes visible. The test results are given as the amount of “mL sand” required to remove a certain thickness of the coating.

Impact strength test: (ASTM D 2794-69). In this test method, the cylindrical steel standards in different weights (1 and 2 kg) are dropped from different heights by a guide tube onto the coated panel. The test is repeated by first increasing the drop height, then changing the standard weight until the coating deforms (cracking, detaching, peeling, etc.). The results are given in “kg x cm” based on the falling weight and height causing the deformation.

Gloss test: (ASTM D 523). In this test method, the gloss value at a certain angle is determined by comparing the specular reflectance from the coating to that from a black glass standard. The results are given as “gloss unit (GU)”. According to the standards, gloss levels determined using a 60° gloss angle were categorized as follows: 0-20 GU is matt, 20-60 GU is semi-gloss, 60-80 GU is gloss, and > 80 GU is high gloss. Therefore, the values higher than 80-85 GU obtained in gloss measurements at 60^o correspond to very glossy coatings (Sönmez 2020, Transocean Coatings 2023).

Chemical surface coating properties

Alkali resistance, acid resistance, salt resistance, and water resistance tests: These tests were done according to ASTM D1647 and ASTM D1308 standards at room conditions (20±2°C, 60±5% relative humidity). In the alkali, acid, and water resistance tests; film-coated glass tubes are immersed in an alkali solution (0.1 M NaOH and 0.01 M NaOH), film-coated glass panels are immersed in an acid solution (3% H_2SO_4, wt.), and film-coated glass panels are immersed in a salt solution (5% NaCl, wt.), respectively. Then the tubes/panels are removed from the solutions after immersion at given time intervals. Then, the appearances of test samples are investigated and compared with the standards, visually. In the water resistance test, film-coated tin panels are immersed in distilled water at room temperature for 18 h. At the end of the 18 h, the appearances of the films are evaluated after being wiped dry, 20 min later, 1 h later, and 2 h later, visually.

Solvent resistance test: This test was performed as given in the literature (ISO 2812-3 and Mizutani et al. 2006). In this test, solvent-impregnated absorbent gauze pieces (1x1 cm) are put on the film-coated glass panel. The panels are covered and kept at room temperature for 30 minutes. At the end of this time, the appearances of films are evaluated visually. In this study, although acetone, methanol, toluene, and ethyl acetate were used for the solvent test, the desired solvent can be used.

Environmental resistance test (wet–cold dry and heat cycle test): This test was performed as given in the literature (Mizutani et al. 2006), and it consists of three steps. *First step:* film-coated glass panel is immersed in a water bath kept at room temperature (20±2°C) for 18 h. *Second step:* the panel is taken out and cooled to -18±2°C in a refrigerator for 3 h. *Third step:* the panel is heated to 50±2°C in an oven for 3 h. This cycle is repeated until a deformation occurs on the film surface. After each cycle, the change of the sample surface in appearance, such as cracking, blistering, or peeling, is inspected visually. Since no change was observed on the film surfaces in this study, the test was ended after the 10 cycles.

Thermogravimetric analyses

Thermogravimetric analyses (TGA) were used to investigate the thermal behaviors and to evaluate the thermal stabilities of alkyd resins. In addition, thermal-oxidative degradation temperatures of the cured resin films were also determined. TGA analyses of alkyd films were carried out in the air atmosphere, with ~10-20 mg sample, by heating from room temperature to 700°C, at a rate of 10°C/min.

RESULTS AND DISCUSSION

Physical coating performances of alkyd resins

Drying behavior

First of all, the drying behaviors of the alkyd resin films were monitored at room temperature for 96 h in the air, and the drying profiles were determined with the drying time test. The drying degrees of all alkyd resins determined by the Modified Bandow-Wolff method in the air are presented in Table VI.

As can be seen from Table VI, the films prepared from reference alkyd resins have dried faster than PET-based alkyd resin films in the air. All reference alkyd resin films, while reaching 1st drying degree in 2 h, PET-based alkyd resin films have reached 1st drying degree in 2-4 h. The drying rates of PET-based alkyd resin films are slower than the reference alkyd resin films, and the final drying degrees they reach after a certain time (after 96 h) are lower. While all reference alkyd films reached the highest drying degree/stage of this test method (7th stage)

Table VI. Drying degrees of all oil-modified alkyd resin films in the air at room temperature according to Modified Bandow-Wolff method.

	Drying degrees (or stages)					
Time (h)	**REF-Alk-100**	**REF-Alk-90**	**REF-Alk-80**	**PET-Alk-100**	**PET-Alk-90**	**PET-Alk-80**
2	1	1	1	-	-	1
2.5	2	2	2	-	-	2
3	2	3	4	-	-	3
3.5	2	3	4	-	1	4
4	2	4	4	1	1	4
24	2	4	5	2	2	4
48	3	5	6	2	3	4
72	7	7	7	3	3	5
96	7	7	7	3	3	6
OC*	7	7	7	7	7	7

***OC: Oven-cured: After oven cured at 120°C for 1 h.**

Table VII. Possible equivalents of drying degrees of all oil-modified alkyd resin films according to Finger-Touch method.

Alkyds	Time to reach dry-to-touch stage*	The possible equivalents of drying degrees according to other standard* (possible drying stages/levels)				After oven cured
Reference alkyds		**24 h**	**48 h**	**72 h**	**96 h**	**120°C, 1 h**
REF-Alk-100	2 h	Surface-dry	Surface-dry	Full-dry	Full-dry	Full-dry
REF-Alk-90	2 h	Dry	Hard-dry	Full-dry	Full-dry	Full-dry
REF-Alk-80	2 h	Hard-dry	Through-dry	Full-dry	Full-dry	Full-dry
PET-based alkyds						
PET-Alk-100	4 h	Surface-dry	Surface-dry	Surface-dry	Surface-dry	Full-dry
PET-Alk-90	3.5 h	Surface-dry	Surface-dry	Surface-dry	Surface-dry	Full-dry
PET-Alk-80	2 h	Dry	Dry	Hard-dry	Through-dry	Full-dry

***Determined by the Finger-touch method (ASTM D 1640). The definitions of all drying terms of this method are given above.**

after 72 h, PET-based alkyd resin films remained at the lower drying stages.

For the purpose of comparison, the possible equivalents of the drying degrees (which determined by the Modified Bandow-Wolff method) according to other standard (Finger-Touch method) are presented in Table VII.

There are two different steps during the drying of alkyds. The first step is *physical drying*, during which the solvent evaporates. In this step, the evaporation of the volatile components takes place. The second stage is *chemical drying*, which involves “oxidation” and “crosslinking reactions”. Thus, this step may also be named oxidative drying. The chemical drying by oxidation is the combination of four steps: “induction period (oxygen uptake)”, “peroxide formation”, “peroxide decomposition into free radicals”, and “polymerization (crosslinking reactions)” (Van Gorkum & Bouwman 2005, Bieleman 2004, 2000, Soucek et al. 2012).

As can be seen, the drying process includes several drying stages involving some physical and chemical changes (i.e., solvent evaporation, reaction with oxygen or moisture, polymerization, or a combination thereof). However, the time to reach these drying stages mentioned here, which is also significantly affected by environmental factors such as film thickness, temperature, and humidity, is challenging to measure. Therefore, the drying stages of coatings are usually defined and determined by whether they correspond to some standard test methods rather than physical or chemical changes. The drying stages, which are difficult to measure and distinguish from each other, can be transformed into comparable results with different measurement and evaluation methods according to tangible definitions of these standardized tests.

As can be seen in Table VI and Table VII, reference and PET-based oil modified alkyd resin films reached the “dry-to-touch” stage in 2-4 h in the air according to the Finger-Touch method. As time progressed, the drying degree (or drying stage or dryness degree) also increased, but the time taken for the drying process became longer. Drying stages of reference alkyd resin films (REF-Alk-100, REF-Alk-90, REF-Alk-80) were determined as 3, 5, 6, and 7, 7, 7 at the end of 48 h and 72 h, respectively. On the other hand, these values for PET-based alkyd films (PET-Alk-100, PET-Alk-90, PET-Alk-80) were determined as 2, 3, 4, and 3, 3, 5 at the end of 48 h and 72 h, respectively.

The “dry-to-touch” stage indicates the time required for the volatile components to separate. From this definition, it is understood that the “dry-to-touch” stage corresponds to physical drying. Therefore, we can say that all alkyd films have completed their physical drying in a short time, and as a result of the evaporation of the solvent, they have formed a film that no longer adheres to the finger upon light touch. These results showed that all alkyd films reached the touch-dry stage relatively quickly. In addition, “stage 3” defined in the Modified-Bandow-Wolff method, which is performed using the load of 200 g, may be considered the possible equivalent of “surface-dry” according to the other standard methods. Similarly, “stages 4, 5, 6, and 7” of this method, performed using loads of 2 kg and 20 kg (20 N and 200 N), may correspond to the “dry, hard-dry, through-dry, and full-dry” and may be compared to each other.

In summary, as seen in Table VII, which shows the possible equivalents of the drying levels of alkyd resins according to the Finger-Touch method, although the drying stages/levels of PET-based alkyd resins are lower than the reference alkyd resins, it is seen as an acceptable/curable level. After 96 h in the air, PET-Alk-100 and PET-Alk-90 resin films reached the “surface-dry” stage, while PET-Alk-80 resin film reached the “through-dry” stage. However, when all PET-based alkyd films were cured in the oven at 120°C for 1 h, they also reached the “full-dry” stage, like the reference alkyd films.

Drying profile and drying mechanism

In the Modified Bandow-Wolff Method, it is determined which drying degree/stage the coatings reach between 1-7 and how long it takes. Therefore, the results obtained in this test enable the evaluation of the drying rate as well as the drying degree/stage and give a rough idea of the drying profile of the coating. The drying profiles of reference and PET-based alkyd resin films determined by this method in the air are presented in Figure 4 and Figure 5.

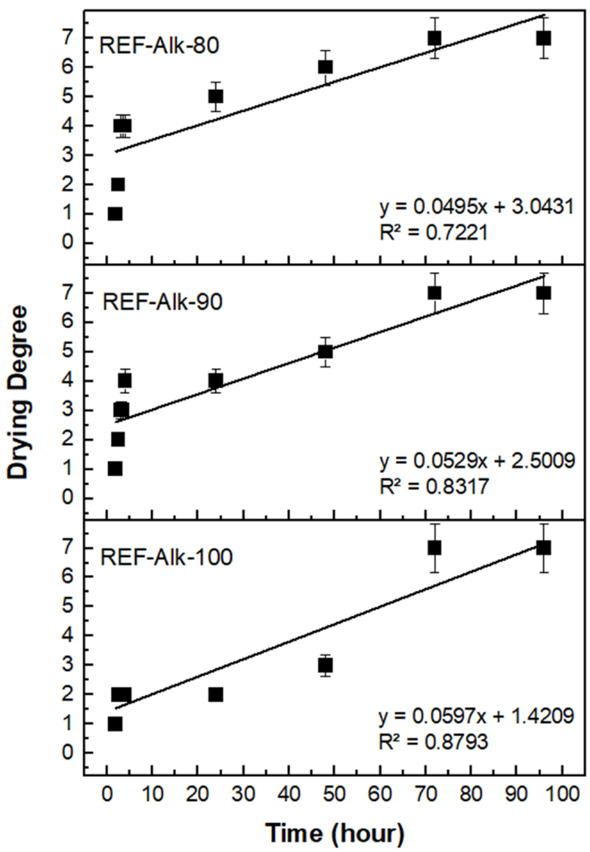


Figure 4. Drying profiles of reference oil-modified alkyd resins in the air.

Although these linear graphs give a rough idea about the drying speed of alkyd films, the drying mechanism of alkyds is quite complex, and there are many factors affecting this mechanism. Therefore, linear analysis methods alone will not be sufficient in modeling drying rates and behaviors. For this reason, it is not surprising that the R-squared values obtained by linear regression remain in the 0.7-0.8 interval. Therefore, the application of measurement methods that can monitor the drying process

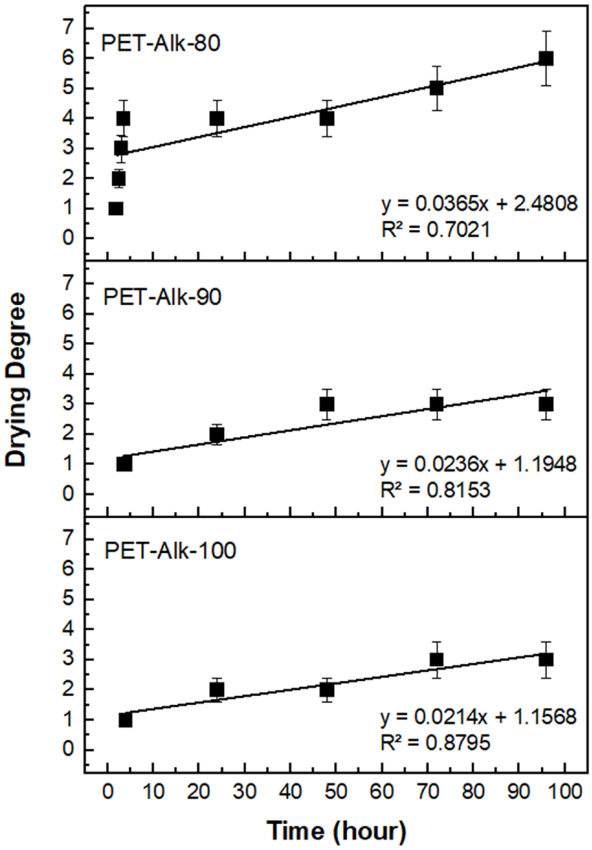


Figure 5. Drying profiles of PET-based oil-modified alkyd resins in the air.

and the influencing factors simultaneously, and multiple data analyses by evaluating all factors affecting the drying behavior separately will provide more accurate results in elucidating the drying mechanism. All factors affecting the drying mechanism in the air of the alkyd resins synthesized here are explained comparatively below.

Effect of depolymerization product (DP) on drying behavior

While reference alkyd films (REF-Alk-100, REF-Alk-90, REF-Alk-80) reached stages 7, 7, 7; waste PET-based alkyd films (PET-Alk-100, PET-Alk-90, PET-Alk-80) remained at stages 3, 3, and 5 after 72 h. This situation probably originated from the higher molecular weight PET depolymerization product having long oligomeric chains, which partially added to the alkyd structure instead of a small phthalic anhydride molecule as the diacid component.

In this study, film formation via cross-linked structure mainly occurred due to oxidative curing of double bonds in the alkyd structure. In this context, the relatively lower molecular weight reference alkyd structure, which is faster motion during the drying, has probably dried slightly faster than the higher molecular weight PET-based alkyd structure having long oligomer chains. It is known that alkyd resins of lower molecular weight dried/cured more easily than alkyd of higher molecular weight (Atta et al. 2013). Further reactions of the remaining double bonds of higher molecular weight alkyd may increase the value of time required for complete drying/curing and decreases the maximum heat evolved upon curing, which indicates that drying occurs more slowly (Atta et al. 2013).

In addition, it is also possible that the condensation reaction of free hydroxyl and carboxyl groups may occur during the drying process (Büyükyonga et al. 2017). The hydroxyl groups in the resin structure can undergo a self-condensation reaction to form ether linkages (Gogoi et al. 2015). Therefore, unsaturated double bonds and free reactive functional groups in the structure will affect this mechanism.

As mentioned above, the drying mechanism of alkyd resin, which is a complex process, takes place in two stages. The first stage involves solvent evaporation, and the second is the oxidative drying of the fatty acid chains. These drying stages eventually result in the formation of a polymer network. As previously reported in the literature, the drying rate and film formation are affected by many factors, such as the unsaturation degree, amount of conjugated double bonds, type and amount of drying catalyst (dryer), molecular weight, molecular weight distribution, crosslink density, and curing conditions etc. Viscosity and glass transition temperature are also important in the film formation stage. As the viscosity and glass transition temperature increase, the free volume decreases, and the solvent evaporation rate depends on how fast the solvent molecules can reach the film's surface. This means that, the rate of solvent loss is controlled by the diffusion rate of the solvent through the film. In this context, PET-based alkyd films containing the higher molecular weight component (PET oligomer) showed a longer drying time than reference alkyd films, possibly due to solvent retention. In other words, during the drying of the high molecular weight fractions by both solvent evaporation and crosslinking, the solvent molecules were probably retained within coating due to the lower solvent diffusivity in relatively high viscosity (Spasojevic et al. 2015, Holmberg 1987, Baghdachi 2007). Also, since long chain PET oligomers chains will also limit chain mobility as well as an increase in molecular weight, the glass transition temperature will increase, and thus the free volume will decrease, possibly resulting in a slower drying rate (Baghdachi 2007).

Effect of LO/CO blend on drying behavior

Although PET-based alkyd resins reached the "dry-to-touch" stage relatively quickly, they have dried for a long time in the air at room temperature. The relatively higher molecular weight and longer chain PET depolymerization product (DP) in alkyd structure caused longer drying time and lower degrees. However, using an LO/CO blend as the monobasic acid component instead of only LO slightly increased the drying speed and drying degree for both reference and PET-based resins.

As can be seen in Table VI and VII, when the LO ratio by weight in the LO/CO blend used in reference alkyds changed in order 100, 90, 80 (REF-Alk-100, REF-Alk-90, REF-Alk-80), it is observed that the drying stages of the alkyd films are progressed in the order of the "surface-dry", "hard-dry", "through-dry", at the end of the 48 h. In these alkyds, although the amount of LO with a higher iodine value (is a measure of the relative degree of unsaturation in oil) decreased the drying rate of alkyds increased. At the end of 72 h, all films reached the full-drying stage.

PET-based alkyd films containing LO/CO blends in the same proportions dried more slowly than reference alkyds, but showed a similar drying tendency with reference alkyds. At the end of the 48 h alkyd films which is remaining in the "surface-dry", "surface-dry" and "dry" stages (PET-Alk-100, PET-Alk-90, PET-Alk-80) were able to reached the "hard-dry" stage at the end of the 72 h. As observed in the reference alkyds, the rate and degree of drying increased in these alkyds, although the amount of LO in the LO/CO blend was decreased.

As known, drying of alkyd resins involves the oxidative processes that require a catalyst (Büyükyonga et al. 2017, Van Gorkum & Bouwman 2005) and they can undergo autoxidation or photo-oxidation, or thermal-oxidation under different conditions with free radicals or oxygen molecules (İşeri-Çağlar et al. 2014, Ofoedu et al. 2021). The autoxidation process is the combination of oxygen uptake, peroxide formation, decomposition of peroxide into free radicals, and polymerization (crosslinking reactions) (Van Gorkum & Bouwman 2005, Bieleman 2004, 2000, Soucek et al. 2012). This autoxidation process begins with the oxygen molecule in the air added into carbon-hydrogen bonds adjacent to the double bonds within the unsaturated fatty acid chains of vegetable oil, and it continues with the crosslinking reactions of hydroperoxides formed (İşeri-Çağlar et al. 2014). Eventually, bonds are formed between the neighbor fatty acid chains, followed by a polymer network formation that can be observed as a film layer (İşeri-Çağlar et al. 2014, Ang & Gan 2012). Therefore, the degree of unsaturation of the resin system plays an important role in affecting the drying or curing rate of the coating (Ang & Gan 2012, Wicks et al. 1999, NIIR Board of Consultants and Engineers 2002). In addition, since the polymerization and cross-linking reactions that provide film formation and drying process will occur through the double bonds, double bonds will significantly also affect the film properties of the coatings at the same time (Ang & Gan 2012, Kickelbick 2007). For these reasons, it is also important which oil was used in the alkyd formulation.

Linseed oil has a high ratio of essential polyunsaturated fatty acids and is prone to oxidation. However, rapid-drying linseed oil which is rich in linolenic acid causes a high degree of residual unsaturation in the cured film. Moreover, rapid drying causes a poor thorough cure due to the formation of a top layer acting as a diffusion barrier to oxygen (Stenberg et al. 2005). In this context, the blending of different oils may be efficiency way for changing their oxidation rate and ratio, and blending linseed oil with other vegetable oils may be change drying time and degree (Golmakani et al. 2020). In this study, such an effect was probably observed in these alkyd resin systems where different ratios of LO/CO blend were used.

Since none of the PET-based alkyd resin films reached the 7th drying degree at the end of the 96 h in the air, all films were oven cured at 120°C for 1 h. And thus, all films exhibited excellent drying properties after being oven cured. Then, all physical (adhesion, impact strength, abrasion resistance, hardness and gloss) and chemical (water, alkali, acid, salt, environmental and solvent resistance) surface coating tests were applied only to the oven-cured films.

Hardness, abrasion resistance, adhesion, impact strength, hardness and gloss properties

Pendulum hardness (König), abrasion wear via falling sand, adhesion strength, impact resistance, and gloss test results of all alkyd resin films are presented in Table VIII.

As seen in Table VIII, in general, glossy films with excellent adhesion, good impact strength and relatively high abrasion resistance were obtained from the PET-based alkyd resins.

In case of reference alkyd resins, the use of LO/CO blend increased the hardness value

Table VIII. Physical surface coating properties of all oil-modified alkyd resins.

Alkyds	Pendulum Hardness (König second)	Abrasion resistance (mL sand)	Adhesion strength (%)	Impact resistance (kg.cm)	Gloss (gloss unit, GU) (at 60°)
Reference alkyds					
REF-Alk-100	106	5000	100	>200	145
REF-Alk-90	132	2000	100	>200	116
REF-Alk-80	80	4000	100	>200	117
PET-based alkyds					
PET-Alk-100	34	3500	100	>200	135
PET-Alk-90	32	4000	100	>200	135
PET-Alk-80	35	4500	100	>200	132

*** The tests were applied to oven-cured films.**

first, but when the ratio of CO in blend was increased from 10% to 20% (by eq. weight), the hardness value decreased due to the decrease in unsaturated bond and cross-linking. Similar behavior was also observed in the gloss property.

However, when the reference and analogous PET-based alkyd resins, which synthesized under the same conditions and containing the same proportions of LO/CO blend, were compared to each other, it was observed that, the use of oligomeric waste PET depolymerization product (DP) with the different ratios LO/CO blends provided softer films. It is seen that the use of PET product in the synthesis of alkyd resin have a positive effect on physical surface coating properties compared with the reference resin. The use of oligomeric waste PET products caused softer, relatively flexible and more glossy films to be obtained.

Chemical coating performances of alkyd resins

Water, alkali, acid, salt, solvent and environmental resistance properties

Water-resistance (distilled water), alkali resistance (0.1 M NaOH and 0.01 M NaOH), acid resistance (3% H_2SO_4, wt.), salt resistance (5% NaCl, wt.), and environmental resistance (wet-cold dry-heat cycle) test results of all alkyd resin films are presented in Table IX. Solvent resistance test results of all alkyd resin films are also presented in Table X.

As seen in Table IX, all oil-modified alkyd resin films have not been affected by acid solution for

Table IX. Chemical resistance test results of all oil-modified alkyd resins.

	Water resistance		Alkali resistance	Acid resistance	Salt resistance	Environmental resistance
	Distilled water		0.1 M NaOH 0.01 M NaOH	3% H_2SO_4	5% NaCl	Wet, cold dry, heat cycle
Alkyds	After wipe dry[a]	20 min, 1 h, 2h later[b]	complete dissolution time[c]	after 72 h	after 72 h	after 10 cycles[d]
Reference alkyds						
REF-Alk-100	T	T	> 2 days > 5 days	NC	NC	NC
REF-Alk-90	T	T	> 2 days > 5 days	NC	NC	NC
REF-Alk-80	T	T	> 2 days > 5 days	NC	NC	NC
PET-based alkyds						
PET-Alk-100	T	T	> 2 days > 5 days	NC	NC	NC
PET-Alk-90	T	T	> 2 days > 5 days	NC	NC	NC
PET-Alk-80	T	T	> 2 days > 5 days	NC	NC	NC

*** The tests were applied to oven-cured films; T: transparent film; NC: no change.**
[a] After being immersed in distilled water for 18 h and wiped dry.
[b] Additional time after immersed in distilled water for 18 h and removed from the water (20 min, 1 h and 2 h later).
[c] The total time it takes for the film to completely dissolve or remove from the surface.
[d] One cycle: in distilled water at room temperature for 18 h + in a deep freezer at -20±2°C for 3 h + in an oven set at 50±2°C for 3 h.

72 h, and all resin films showed excellent acid resistance. The salt resistance test performed with 5% NaCl solution also gives an idea about the corrosion resistance of the coating, and the corrosion resistance of all oil-modified alkyd films is also excellent after 72 h. According to the standard of the water-resistance test, the films are examined visually at the end of the test, and the results are given as whitening or clouding or remaining transparent of the film. As seen in Table IX, all resin films remained transparent, and there was no change on the surface of the films after 18 h. Both the reference and PET-based alkyd resin films were not affected by distilled water at room temperature. The use of PET depolymerization product (DP) in alkyd resin formulations did not cause a negative effect on water resistance property already perfect, and all resin films showed excellent water resistance.

At the end of the wet-cold dry and heat cycle testing, which simulates environmental conditions at a small scale and allows multiple repetitions, it was observed that the changing environmental conditions did not cause any effect on the films. Even after 10 cycles, it is seen that the environmental resistances of all alkyd resin films were excellent (Table IX). The use of waste PET product did not change the environmental resistance of the resins.

When examining the alkali resistance test results (Table IX), the complete dissolution times (the total time it takes for the film to dissolve or remove from the surface completely) of all alkyd resin films in 0.1 M NaOH solution were observed as more than 2 days. In reference alkyd resin films, the first effect arise from 0.1 M NaOH solution was observed after 24 h, whereas in PET-based alkyd resin films, the first effect was observed within the first 10 h. However, complete dissolution of all reference and PET-based resin films occurred after 48 h.

In addition, when the alkali resistance test was repeated with 0.01 M NaOH solution, all the alkyd resin films were not dissolved even after 5 days and the films remained intact on the test plates. It is known that the alkali resistance of alkyd resins is weak due to the content of hydrolyzable ester bonds, and the rate of hydrolysis reactions increases even more under alkaline conditions (Issam et al. 2011). Therefore, for alkyd resins which are known to have weak alkali resistance, obtained results using diluted NaOH solutions in this study are at acceptable levels and appear to be suitable for improvement.

Table X. Solvent resistance test results of all oil-modified alkyd resins.

Alkyds	Solvent resistance*			
	Acetone	Toluene	Methanol	Ethyl acetate
Reference alkyds				
REF-Alk-100	NA	NA	ST	ST
REF-Alk-90	NA	NA	ST	ST
REF-Alk-80	NA	NA	ST	ST
PET-based alkyds				
PET-Alk-100	NA	NA	ST	ST
PET-Alk-90	NA	NA	ST	ST
PET-Alk-80	NA	NA	ST	ST

*** The tests were applied to oven-cured films. NA: not affected, ST: slight trace.**

In solvent resistance test; methanol, toluene, acetone, and ethyl acetate were used as a solvent. At the end of this test, some resin films had slight traces, but no dissolution or significant surface deformation was observed in any of the films (Table X). Therefore, we can say that the solvent resistance properties of all alkyd resins were close to excellent.

Thermal analysis results

TGA thermograms of all reference and PET-based alkyd resin films are presented in Figure 6 and Figure 7, respectively. In addition, “temperature - weight loss” table showing the temperatures corresponding to certain weight losses were also presented in Table XI.

In general, the thermal behavior profiles of all alkyd resins modified with the LO/CO blend are similar. However, PET-based alkyd resins have higher thermal resistance than reference resins.

The temperature values corresponding to certain weight losses of the reference alkyd resins are very close to each other up to 50% weight losses. After 50% weight loss, although the temperatures are close to each other, small fluctuations are observed. In addition, the temperature values of PET-based alkyd resins corresponding to certain weight losses are very close to the reference resins up to 50% weight losses. However, after 50% weight loss, it was observed that the thermal resistance of PET-based resins increased. For example, reference resins reached 50% weight loss at around 313-232°C, while PET-based resins reached 50% weight loss at around 327-336°C.

Final thermal oxidative degradation temperatures were observed as 468°C and 495°C for REF-Alk-100 and REF-Alk-80 resins, respectively, for 90% weight loss. For PET-Alk-100, PET-Alk-90, and PET-Alk-80 resins, these values were observed as 572°C, 525°C, and 529°C, respectively. As can be seen from these results, in general, PET-based oil-modified alkyd resins have significantly higher thermal resistance than reference resins. As a result, it is seen that the use of waste PET depolymerization product in the formulation of LO/CO blend-modified alkyd resins did not cause any negative effects on thermal strengths. On the contrary, it significantly

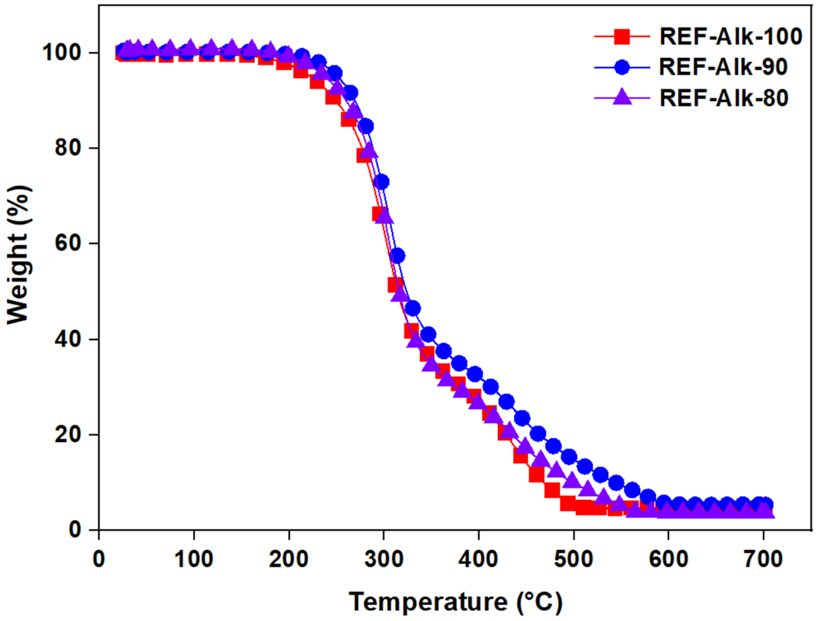


Figure 6. TGA thermograms of reference oil-modified alkyd resins.

increased the final thermal oxidative degradation temperatures of the resins compared to the reference resin. Presumably, this is due to the introduction of relatively long-chain/aromatic oligomeric units into the structure. The use of waste PET depolymerization product in the alkyd resin formulation increased the final thermal oxidative degradation temperatures, that is, increased thermal stabilities of the resins.

Another approach is the effect of waste PET oligomers as well as the LO/CO blend on thermal-oxidative stability. The blending of vegetable oils has been tried as an effective approach to improve their oxidative stability. As given in the literature previously, linseed oil which is a rich source of polyunsaturated fatty acids and has poor stability against oxidation has been blended with different vegetable oils to enhance its oxidative stability. In these mentioned studies, blending linseed oil with other vegetable oils improved oxidative stability and reduced the dependency of the oxidation rate on temperature (Golmakani et al. 2020). This mentioned approach has been observed in REF-Alk-90 and REF-Alk-80 reference resin films without PET.

As a result, in the case of reference alkyd resins without waste PET, the use of LO/CO blend in formulation has increased thermal stability. In the case of waste PET-based alkyd resins, glossy and soft films with excellent coating properties were obtained by incorporating of the waste PET product (DP) in the formulation at a small ratio of 10% (by wt.) without compromising the superior properties of the alkyd resin. The addition of LO/CO at the ratio of 80/20 (by wt.) to the formulation of these alkyd resins also increased the thermal resistance compared to the reference alkyd resin (REF-Alk-100) without both waste PET product (DP) and LO/CO blend.

In this working range, the optimum LO/CO ratio for waste PET-based alkyd resins was determined as 80/20 (by wt.) in terms of best coating properties and thermal resistance. Waste PET-based alkyd resin film (PET-Alk-80) prepared at this ratio reached the “hard-dry” and “through-dry” stages in the air after 72 and 96 h, respectively. Soft and glossy films having

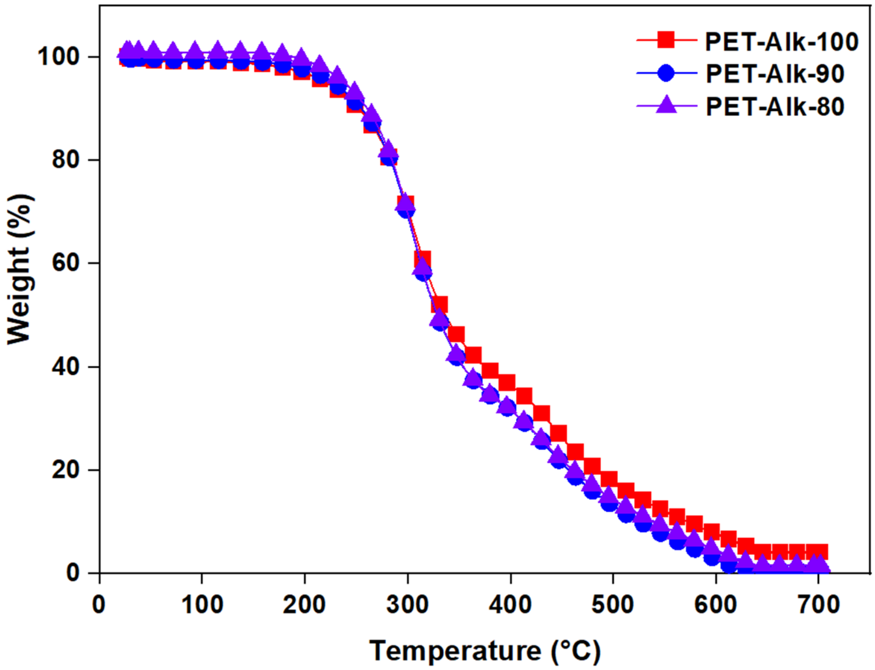


Figure 7. TGA thermograms of PET-based oil-modified alkyd resins.

Table XI. Temperature-weight loss table of all oil-modified alkyd resins.

Weight loss, %	The temperatures corresponding to certain weight losses (ºC)					
	REF-Alk-100	REF-Alk-90	REF-Alk-80	PET-Alk-100	PET-Alk-90	PET-Alk-80
10	250	268	258	252	255	257
20	277	288	282	283	283	283
30	291	300	295	301	299	298
40	303	310	305	316	312	311
50	**313**	**323**	**315**	**336**	**329**	**327**
60	334	349	331	375	354	351
70	382	410	371	434	409	403
80	429	461	432	484	456	455
90	**468**	**540**	**495**	**572**	**525**	**529**

excellent adhesion property, high impact and abrasion resistance, high chemical (solvent) and thermal resistance with resistance to environmental (wet-cold dry-heat cycle) and corrosive conditions (acid, salt, water) were obtained from oven-cured PET-Alk-80 alkyd resin.

Future Perspectives

In this study, it was attempted to determine the alkyd formulation with good surface coating and thermal properties and the optimum ratios for LO/CO blend-modified alkyd resins containing a certain amount of waste PET depolymerization (aminoglycolysis) product. The first results detailed presented above were obtained in this regard. In this study in which many parameters are effective, it will be possible to reach more accurate results by increasing the number of possibilities and combinations. For this purpose, in future studies, it is envisaged to use statistical experimental design methods that allow more parameters to be evaluated with fewer experiments and their effects on the process to be examined accurately and effectively. In four-component alkyd resin formulations having desired surface coating performance and good thermal oxidative stability, the use of different PET depolymerization (such as hydrolysis, glycolysis, aminolysis) products (monomers or oligomers) having different ratios of functional groups that can be substituted (partially or completely) instead of main materials (diacid or diol) and, the use of LO/CO blends (or different oil blends) at changing ratios, together, it seems possible to prepare more effective, applicable, efficient, sustainable, environmentally friendly and economical alkyd resins. This way, it may be possible to recycle post-consumer PET bottles and thus reduce the total amount of plastic waste, thereby important results can be obtained from an economic and environmental point of view. It is anticipated that the findings of future comprehensive studies, together with the results obtained in this study, will contribute to the scientific literature, the development of the paint industry, and waste management studies.

CONCLUSION

Waste PET-based oil-modified alkyd resins were synthesized using PET depolymerization product (DP, functional oligomer mixture obtained from aminoglycolysis reaction of waste PET bottles), and different ratios of linseed oil/canola oil (LO/CO) blend. Then, their coating properties and thermal stabilities were examined comparatively and their suitability for coating applications was evaluated. The effect of the use of DP and LO/CO blend on the coating properties and thermal behaviors of the resin films was investigated. The results obtained in this study are as follows:

LO/CO blend as a monobasic acid component at ratios of 100/0, 90/10, 80/10 (by wt.), and DP as a dibasic acid component instead of the PA at the ratio of 10% (by eq. wt.) was successfully incorporated into the alkyd resins formulations. LO/CO blend and DP were compatible with other alkyd components, and alkyd synthesis reactions were performed without any problems. The glossy, soft/medium-hard films with excellent adhesion, impact resistance and relatively high abrasion resistance were obtained from LO/CO blend-modified alkyd resins synthesized by the monoglyceride method. Although dry-to-touch time is relatively short, the long chain oligomeric DP with high molecular weight probably caused longer drying time and lower drying degree of PET-based alkyd compared to reference alkyd, in the air. Blending different oils and using these blends in the alkyd formulations were effective in changing the oxidation rate and ratio of the oil component, thus changing the drying time and degree of alkyd films. In reference alkyd resins, when the CO ratio in the oil blend was changed, the hardness value also changed due to the change in the unsaturated bond and cross-linking ratios, whereas such an effect was not observed in PET-based alkyd resins. The use of DP in the synthesis of PET-based alkyd resin did not cause any negative effect on physical surface coating properties compared with the reference resin. On the contrary, in PET-based alkyd resins, the use of oligomeric DP with the different ratios of LO/CO blends provided softer and more glossy films. All reference and PET-based LO/CO blend-modified alkyd resin films showed excellent resistance to corrosive conditions (acid, salt, water) and environmental conditions, and they have a great extent of solvent resistance. The complete dissolution times of all alkyd resin films in diluted alkali solution were more than two days, and these results are quite good and seem suitable for improvement. The use of DP in the PET-based alkyd resin formulation increased the final thermal oxidative degradation temperature compared to the reference resin. The increase in thermal stability is due to the incorporation of long-chain and aromatic oligomeric PET units into the alkyd structure. In reference alkyd resins, using of LO/CO blend in the formulation affected the thermal-oxidative stability. Blending linseed oil with canola oil probably improved oxidative stability probably due to the changing double-bond ratio. The optimum LO/CO ratio for PET-based alkyd resins was determined as 80/20 (by wt.) in terms of best coating properties and thermal resistance.

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