https://doi.org/10.3311/PPch.40970

Periodica Polytechnica Chemical Engineering, 69(4), pp. 586–611, 2025

Aldehyde-modified Alkyd Resins Based on Waste Poly(ethylene terephthalate) Prepared by *in situ* Reaction Method for Sustainable Versatile Coating Systems

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Received: 09 May 2025, Accepted: 05 November 2025, Published online: 09 December 2025

Abstract

This study aims to develop novel aldehyde-alkyd systems for versatile coating applications from a four-component alkyd resin containing waste poly(ethylene terephthalate) (PET) using a sustainable approach. Post-consumer bottle flakes were depolymerized *via* a simultaneous hydrolysis-glycolysis reaction. PET-based and reference unmodified alkyd resins were synthesized with and without PET. In the PET-based alkyd formulation, the PET depolymerization product (DP) was used in place of the diol component of alkyd. To produce the modified alkyd resins, the aldehyde resin was incorporated into the reaction medium during the synthesis of the PET-based and reference alkyd resins. Thus, both unmodified and modified alkyds with and without PET were prepared for comparative analysis. The research focused on the effects of aldehyde resin usage and the presence of DP on the coating properties and thermal behavior of the modified alkyd films. The physical/chemical coating properties of both modified and unmodified resins were reasonably sufficient for applications. The hard films obtained from alkyds exhibited superior adhesion strength, excellent impact resistance, and high gloss. Moreover, they were unaffected by water and exhibited remarkable resistance to corrosive agents. Organic solvents did not adversely affect these films. All films demonstrated excellent performance in repeated environmental resistance tests simulating various climatic conditions. The coating and thermal properties of PET-based alkyd films were slightly superior to their reference counterparts due to the incorporation of DP, an oligomer mixture containing aromatic units. Furthermore, *in situ* aldehyde modification slightly increased alkali resistance and thermal stability and reduced gloss loss in PET-based alkyds.

Keywords

poly(ethylene terephthalate), alkyd resin, aldehyde resin, *in situ* modification, waste, recycling, coating, thermal properties

1 Introduction

Sustainability is one of the most critical concepts of today. Green chemistry, clean production, and pollution prevention are crucial to environmental sustainability. In this context, comprehensive evaluations and studies are carried out on ecological principles such as renewable resources, resource minimization, source reduction, waste minimization, recycling, reuse, repair, regeneration, recovery, remanufacturing, and purification [1]. In line with these principles, reducing waste through recycling/recovery and reusing recycled products/raw materials in manufacturing plays an essential role in minimizing environmental impacts using existing resources.

Nowadays, the increasing prevalence of plastic materials in our daily lives, along with urbanization, economic development, and a growing population, leads to a rising amount of plastic waste worldwide [2]. In 2020, a study found that around 150 million tons of plastic were produced each year, with this number continuing to rise [3]. It is reported that the annual production of plastic products increased from 1.5 million tons in the 1950s to 450 million tons in 2024 [4]. However, 79% of this vast amount of plastic produced ends up in landfills, and as a result, 2.41 million tons of these durable waste materials enter the ecosystem every year [3, 5].

Despite the significant amount of waste produced and its negative impact on the environment, the use of plastic materials continues to grow daily due to their advantageous properties, cost-effective production processes, and the convenience they provide in many aspects of daily life.

Today, poly(ethylene terephthalate) (PET) is one of the most widely used plastics. PET, a semi-crystalline

Cite this article as: Erol, T., Acar, I. "Aldehyde-modified Alkyd Resins Based on Waste Poly(ethylene terephthalate) Prepared by *in situ* Reaction Method for Sustainable Versatile Coating Systems", Periodica Polytechnica Chemical Engineering, 69(4), pp. 586–611, 2025. https://doi.org/10.3311/PPch.40970

thermoplastic polymer, is extensively used in the production of bottles (mostly water and soft drink bottles) and fibers due to its high strength, good abrasion and heat resistance, high chemical resistance, excellent dimensional stability, transparent appearance, and low gas permeability [6]. It is also used in food packaging materials, housewares, electronics, sporting goods, photographic films, and various engineering products [7, 8].

Bottles and fibers are key players in the PET market, representing 30.3% and 63.5% of total global PET production, respectively. Their dominance highlights the continuing demand for these materials [9]. In 2016, global usage of PET packaging, excluding fiber, reached 21.7 million tons. Moreover, 87.4% of bottled water sold in Europe that year was packaged in PET [10]. However, the intensive use of PET, which is resistant to atmospheric and environmental conditions, brings an increasing waste problem. PET waste accounts for approximately 12% of the total volume of solid waste generated globally, and this volume is increasing at a concerning rate [11]. According to current projections, global PET demand reached 22.65 million tons in 2025 [5]. PET consumption is estimated to reach 42 million tons by 2030, accounting for 54% of total synthetic fibers and 30% of all bottles produced globally [7]. In addition, market forecasts predict that the worldwide PET market is expected to achieve a compound annual growth rate (CAGR) of 6.7% during the 2022–2030 period, exceeding USD 72.88 billion in revenue by the end of 2030 [9]. Alarmingly, nearly 97% of plastic packaging is disposed of after a single use [9, 12, 13]. Unfortunately, a study conducted in 2019 indicated that only 28.4% of the total production of PET was subject to recycling [14].

These statistical data show that the high consumption of PET leads to significant amounts of post-consumer waste, creating a serious environmental threat. Since the late 1980s, when concerns about plastic waste first emerged, efforts have been focused on recycling post-consumer PET into new value-added products to address this challenge, and these efforts continue to this day.

In this context, for environmental sustainability, determining effective management strategies for plastic waste is necessary to ensure suitable plastic waste disposal and, thus, a clean environment [15]. For this purpose, there are currently physical/mechanical (primary and secondary recycling), chemical (tertiary recycling), and thermal recycling (quaternary recycling) processes used for waste plastics, including PET. In general, these processes include converting plastic waste into secondary materials or raw materials that can be integrated into the system for reuse or to create new valuable products with comparable properties [2, 15]. These methods are explained in detail below [5, 7–9, 11, 16–22].

Primary recycling involves adding clean and uncontaminated PET waste to fresh raw materials in specific ratios during production. However, the formation of hydroperoxide and acetaldehyde often results in colored products with low mechanical properties.

Secondary recycling is defined as the reuse of contaminated plastic waste to produce lower-quality new products. Generally, polymeric waste is reprocessed into granules by conventional extrusion after separation from associated contaminants. The main disadvantage of this mechanical recycling process is the deterioration of product properties, mainly due to the decrease in molecular mass after each cycle as a result of chain breaks.

Tertiary recycling is a recycling method for the production of chemical raw materials from PET waste. The typical chemical reactions used for this purpose are hydrolysis, alcoholysis, glycolysis, and aminolysis.

Quaternary recycling involves utilizing the heat released from the incineration of PET waste to convert it into various forms of energy. However, incineration of PET waste, which has the potential to be used in many different ways, is both economically and environmentally undesirable.

Among all these recovery/recycling methods, chemical recycling is a generally accepted technique due to its product quality, economy, sustainability, and environmental friendliness.

Chemical recycling is applied in the form of degradation of polymeric bonds to convert waste plastic materials into small molecules [23], which are then used as raw materials to manufacture valuable products [24]. Various studies use this approach to obtain functional products (monomer, dimer, oligomer, etc.) as a result of depolymerization reactions of waste PET bottles. These products are then used partially or completely instead of raw materials in the synthesis of various resins such as solvent and water based alkyd resins [25–27], epoxy resin [28, 29], polyurethane resin [30, 31] and/or in paint/coating formulations [32–36]. Among these, the use of waste PET products in alkyd resin production draws attention not only for its environmental benefits but also for its economic gains. Moreover, with various modifications, alkyd resins containing PET can be obtained with the same and/or sometimes superior properties to standard alkyds.

Alkyd resins are synthetic polyester resins obtained by esterification of polyhydroxy alcohols with polybasic acids and fatty acids [37]. Today, alkyd resin-based coating materials are preferred in various areas and versatile applications due to their superior properties [38]. In addition, alkyd resins can be modified in multiple ways to enhance their existing properties and to introduce desired ones [37, 38]. The modification process of alkyds can be carried out by physical (blending) or chemical (*in situ* reaction) methods [39, 40]. The physical modification process is carried out by preparing blends of modifying chemicals with alkyd resin in various ratios [39]. The chemical modification process is done by adding modifying chemicals to the reaction medium during alkyd resin synthesis [40]. There are several studies in the literature on the modification of alkyd resins with various resins such as acrylic [41, 42], urethane [31, 43], epoxy [44, 45], amino [46–49], styrene [50, 51], silicone [52, 53], ketone [54–59], etc. Nevertheless, there are very few studies on modifications made with aldehyde resin [59–61].

Aldehyde resin is a condensation polymer in the low molecular mass varnish resin class. It is a condensation product of urea (or urea derivative) and one or more aliphatic aldehydes. Urea-aldehyde polycondensates are obtainable by the step-wise reaction of urea and/or urea derivatives with aldehydes, and they are usable as surface-coating binders. Due to its high solubility and compatibility, it is widely used in different areas. It improves properties such as hardness, adhesion, gloss, resistance to yellowing, water resistance, pigment wetting, and heat resistance. Since it is very heat-stable, it can be used on baking finishes. It is also used in industrial, metal, automotive paints, and wood coating systems [61–64].

Studies on the very few urea-aldehyde and alkyd combinations in the literature for coating applications are as follows in the following paragraphs.

In the patent study conducted by Gutkin, the urea-aldehyde condensation product resin was first reacted with polyhydric alcohol and then with polybasic carboxylic acids. An esterification reaction of this reaction product with monocarboxylic acids was then carried out. As a result of the study, a viscous and transparent urea-aldehyde-alkyd product resin was obtained, which is used as an adhesive and plasticizer for nitrocellulose resins [60]. In a patent study by Fischer et al. [61], urea-aldehyde polycondensate was added to paint formulations with long-oil alkyd resins, not exceeding 20% of the total formulation. High gloss and yellowing-resistant films were obtained with these formulations applied to wood and metal surfaces. In addition to these two old patents, we also have a recent study of aldehyde-modified alkyd systems prepared by blending long-oil alkyd and aldehyde resins at different ratios [59].

As a result, the preparation of the *in situ* aldehyde-modified alkyd resin presented in this study differs significantly from the studies in the literature. Waste PET depolymerization products (DP) are not used in the mentioned patent studies [60, 61], and their methods are different from this study. In our recent study [59], waste PET was used, but the modification was made using the blending method (physical modification). In these respects, the *in situ* aldehyde-modified alkyd systems with or without PET, which were prepared *via* chemical modification in our study, are novel and entirely different from those in the literature.

This study aims to prepare aldehyde-modified alkyd resin systems that can be employed to obtain sustainable coating formulations by recycling and reusing waste PET bottles as raw materials in production, thereby reducing existing waste. To the best of our knowledge, no detailed experimental study has yet been reported in the literature regarding the *in situ* modified four-component, medium-oil, tall oil fatty acid (TOFA)-based alkyd resin prepared with waste PET DP and aldehyde resin. Within the scope of the study, PET-based *in situ* aldehyde-modified alkyd resin was prepared by using waste PET DP (hydroxyl and carboxyl-terminated PET oligomer) entirely in place of the diol component of the alkyd resin, and partially instead of the dibasic acid component. For comparison purposes, unmodified PET-based alkyd resin, as well as modified and unmodified PET-free alkyd resins, were also prepared similarly. Then, the coating and thermal properties of waste PET-based *in situ* aldehyde-modified alkyd resin were investigated together with the other alkyds prepared for comparison.

To emphasize, this study differs from other studies of aldehyde-modified alkyd systems because it focuses on the *in situ* modification of alkyd resin (with or without PET) with aldehyde resin for the first time.

The advantage of this study is the production of hard, durable coatings with improved properties, including excellent impact resistance, strong adhesion, high gloss, superior chemical resistance, and enhanced thermal resistance. Another advantage of this study is the innovative use of recycled PET in these *in situ* aldehyde-modified alkyd systems.

Finally, this initiative could contribute to the development of cost-effective, durable waste-derived coatings that can compete with traditional alkyd resins used in oven-cured coating applications, while simultaneously reducing existing waste by recycling PET in accordance with sustainability and environmental principles.

The methodology and flow chart of this study are presented in Fig. 1.

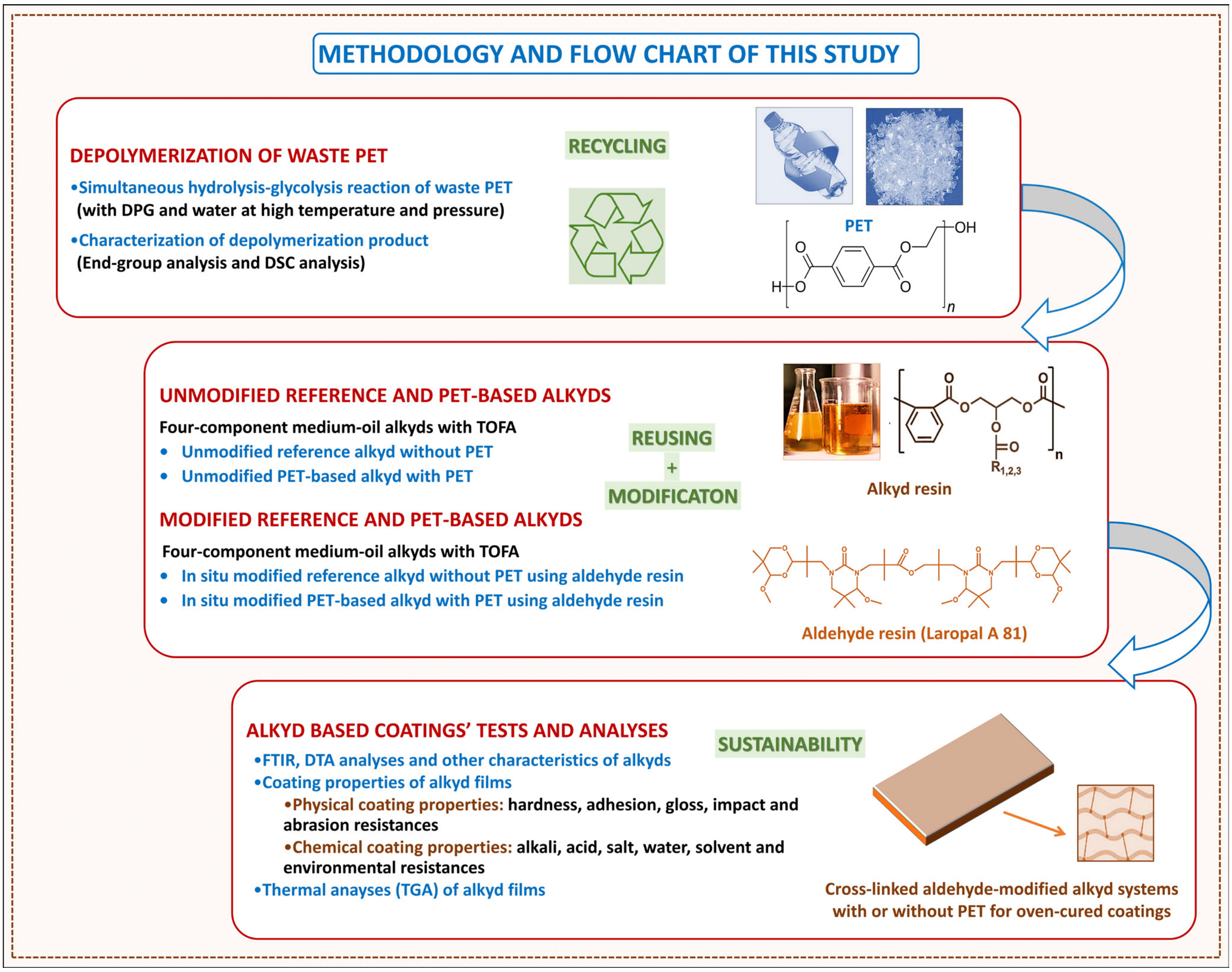


Fig. 1 The methodology and flow chart of this study

2 Materials and methods

2.1 Materials

Waste PET flakes, sieved 8–10 mesh (2.38 mm – 2 mm), were obtained from disposable white and transparent water bottles. The viscosity average molecular mass (M_v), intrinsic viscosity (η), and dispersity (PDI = M_w/M_n = mass average molar mass/number average molar mass) of PET bottles have been previously reported as 37,000, 0.7 g/dL, and around 2–2.5, respectively [33, 59, 65].

TOFA (Sylfat 2S) was provided by Arizona Chemical Ltd. (a Kraton company, USA). The physical properties of TOFA are as follows: acid value (AV) is 193–196 mg KOH/g, saponification number is 197 mg KOH/g, and iodine number is 152 g I/100 g.

Phthalic anhydride (PA) and glycerine (GLY) were Sigma-Aldrich products (USA), and dipropylene glycol (DPG) was a Merck product (Germany).

Aldehyde resin (Laropal A 81, urea-aldehyde resin) was procured from BASF (Germany). It is a condensed product from urea and aliphatic aldehydes [62]. It is formed through a condensation reaction between urea, formaldehyde, and another aliphatic aldehyde (isobutyraldehyde) [63]. The physical properties of this resin [62] are as follows: AV is ≤ 3 mg KOH/g, hydroxyl value (HV) is ~40 mg KOH/g, saponification value is ~65 mg KOH/g, and iodine number is ≤ 3 g I/100g.

Drying agents (6% zirconium octoate, 6% manganese octoate, and 24% lead octoate) were supplied by AKPA Chemicals (Türkiye). All other materials were of synthesis/analytical or technical grade quality.

2.2 Depolymerization of waste PET

Waste PET flakes were depolymerized at high temperature and pressure through a simultaneous hydrolysis-glycolysis reaction conducted within a BR-1000 high-pressure reactor (autoclave) (Berghof, Germany).

This reaction was carried out with a 1/1 molar ratio of PET to diol, using DPG and 60 mL of water, with zinc acetate as a catalyst at a ratio of 1% by mass of PET. The reaction occurred in the presence of 250 mL xylene at a controlled temperature of 225 °C (±5 °C accuracy) and pressure (25–26 bar), for 3 h. The amount of reactants was calculated based on the repeating unit mass (molar mass) of PET, which is 192 g/mol. After the reaction, the resultant raw DP was decanted to remove the xylene phase. It was dried in a vacuum oven at 60 °C without any additional purification, and it was ground in an agate mortar to form a powder.

2.3 Characterization of the depolymerization product

To characterize the DP, AV (ASTM D1639-90(1996)e1 standard [66]) and HV (ASTM E222-10 standard [67]) analyses were performed, and the theoretical number average molecular mass (M_n) of DP was calculated. Additionally, differential scanning calorimetry (DSC) measurement was conducted. Analysis conditions are specified in the relevant sections.

2.4 Synthesis of unmodified alkyd resins

Firstly, the syntheses of an unmodified reference alkyd resin without PET (REF-ALK) and an unmodified waste PET-based alkyd resin (PET-ALK) were carried out to compare with *in situ* aldehyde-modified resins with and without PET.

Synthesis of alkyd resins was carried out in a glass reactor system using the fatty acid method. This glass reactor system consists of a five-neck glass reactor, a mechanical stirrer with a Teflon stir rod and blade, a gas inlet, a contact thermometer, a reflux condenser, a Dean-Stark unit, and a heating mantle. The components used for alkyd resin synthesis and the catalyst were weighed and loaded into the five-neck glass reactor, and then xylene was added. The reaction system was gradually heated under continuous stirring in a nitrogen atmosphere. The water released during the reaction was collected in the Dean-Stark part by forming an azeotrope with xylene. The progress of the reaction was monitored by measuring the AV of the samples taken from the reaction medium at specific intervals, starting from the moment when the first water output was observed. After the temperature reached 220 °C, the reaction was continued until the desired AV was achieved.

2.5 Synthesis of *in situ* aldehyde-modified alkyd resins

Chemical modification reactions of alkyd resins with aldehyde resin were carried out in the glass reactor system used in alkyd resin synthesis. Aldehyde resin (Laropal A 81), which is used as a modifier was added *in situ* to the reaction medium during the alkyd resin synthesis. The amount of aldehyde resin was 7% by mass of the total initial charge of alkyd resin and diluted with 50% xylene by mass. The addition of Laropal A 81 was executed slowly and gradually during the last stage of the reaction process. The AV was determined by taking samples from the medium before and after adding aldehyde resin to the reaction system.

2.6 Characterizations of alkyd resins

Characterizations of both unmodified and aldehyde-modified alkyd resins were conducted using several methods, including Fourier transform infrared spectroscopy (FTIR), differential thermal analysis (DTA), DSC, and thermogravimetric analysis (TGA). The viscosity of the synthesized alkyd resins was also measured. The analysis conditions are detailed in the relevant sections.

2.7 Preparation of alkyd resin films

To perform physical and chemical surface coating tests of resins, resin films were prepared on different surfaces (glass plate, metal plate, tin plate, glass tube) using various methods (with applicator, casting technique, dipping technique) in accordance with the relevant standards. For this purpose, firstly, unmodified and *in situ* modified alkyd resins were diluted to 60% solid content with xylene. Then, driers were added in amounts to contain 0.1% Mn, 0.2% Zr, and 0.39% Pb by mass according to the amount of alkyd resin. Following this, according to the relevant standards, resin films were prepared on glass and metal plates of 10 × 15 cm (width × length) with a 50 μ BYK applicator, on glass tubes of 3 × 12 cm (diameter × height) with a dipping technique, and on tin plates of 5 × 10 cm (width × length) with a casting technique.

2.8 Coating tests of alkyd resin films

To determine the physical and chemical coating properties of unmodified and *in situ* modified alkyd resin films, standard surface coating test methods were applied. These tests are listed below.

2.8.1 Physical surface coating tests

The physical surface coating tests (drying degree, hardness, adhesion strength, abrasion resistance, impact resistance and gloss) conducted in this study are summarized below, with references to relevant standards [68, 70, 72–76]:

- *Drying degree (DIN 53150:1995-06* [68], *Modified Bandow-Wolf method, Erichsen 415/E Drying Tester)*: the drying degree (or stage) is rated from 1 to 7 in this standard [68]. These stages are determined by the adherence or non-adherence of glass beads or craft paper to the coating. For the 1st stage, the glass beads are used without applying a load. For the 2nd to 7th stages, specific loads (ranging from 5 to 5000 g/cm^2) are applied for 60 s. The drying degrees of the films are measured at 20 ± 2 °C, at 60 ± 5% relative humidity [69].

- *Hardness (DIN 53157:1987-01* [70], *Sheen 707 Pendulum Hardness Rocker)*: König hardness is determined by measuring the mechanical damping of a pendulum oscillating on the coating within the specified amplitude limits (from 6° down to 3°). The damping-off or a decrease in the amplitude of an oscillation time of the König pendulum on the standard glass surface, which is the hardest material for this test, is 250 s [71].
- *Adhesion strength (ASTM D3359-23* [72] *and ISO 2409:2020* [73], *Erichsen GS 10 Cross-Cutter)*: in the adhesion strength test, cuts at right angles to each other (square-shaped lattice pattern) are created on the coating surface on a glass panel with a 6-blade cross-cutter. Following this, the surface is brushed, or the tape is applied to the surface and then removed, and the resultant pattern is compared with images in the standards [72, 73].
- *Abrasion resistance (ASTM D968-05* [74], *Erichsen 2511-11 Falling Sand Abrasion Tester)*: for the abrasion resistance test, abrasive hard sand is poured through a vertical tube onto the coating on a glass panel placed at a 45° angle. The volume (mL) of sand that erodes a specific coating thickness (creating a gap of about 4 mm in diameter) is determined.
- *Impact resistance (ASTM D2794-93(2024)* [75], *BYK Gardner PF-1115 Light-Duty Falling Weight Impact Tester)*: in the impact resistance test, a standard steel (1 or 2 kg) load (mass) is dropped through a guide tube onto a coating on a metal plate from varying heights (maximum 100 cm), and the deformation caused by the falling mass and height is measured (kg × cm).
- *Gloss (ASTM D523-25* [76], *Sheen 101N mini single-angle Gloss Meter)*: the principle of this test is based on measuring the ability of the coating surface to reflect an incident beam of light at a certain angle and fixed intensity. The device measures the amount of reflected light from the coating surface. Gloss at a specific angle (in this study, at 60° angle) is determined by comparing the specular reflectance of the coating with that of black glass. The result is given as a gloss unit (GU). According to the Master Painters Institute (MPI), values between 70 and 85 GU measured at a 60° angle are defined as glossy, while values greater than 85 GU are described as very glossy [77].

2.8.2 Chemical surface coating tests

The chemical surface coating tests (alkali resistance, acid resistance, salt resistance, water resistance, solvent resistance, and environmental resistance) conducted in this study are summarized below, with references to relevant standards [78–81]:

- *Alkali, acid, salt, and water resistance (ASTM D1647-89(1996)e1* [78], *immersion method)*: in the alkali resistance test, alkyd films were immersed in an alkali solution (0.1 M NaOH) and examined visually at specific intervals. In the acid and salt resistance tests, alkyd films were immersed in 3 wt.%. H_2SO_4 and 5 wt.%. NaCl solutions, respectively. Then, they were removed from the solutions at the end of 72 h for the acid resistance test and 120 h for the salt resistance test, and examined visually. In the water resistance test, alkyd films were immersed in distilled water at room temperature (20–25 °C) and kept for 18 h. The films were examined immediately after being removed from the water and wiped dry, and after the following 20 min, 1 h, and 2 h.
- *Solvent resistance (ISO 2812-3:2019* [79], *absorbent medium method)*: in this test, a 1 × 1 cm gauze was immersed in various solvents, including acetone, toluene, methanol, ethyl acetate. After excess solvent was removed, the gauze was placed on the surface of an alkyd film, which was then covered with a petri dish. After allowing the setup to sit for 30 min the surface was examined visually.
- *Environmental resistance/durability (ISO 9211-3:2024* [80] *and ISO 9022-2:2015* [81], *wet-cold-dry-heat cycle and slow temperature change methods)*: this test was conducted as outlined by Mizutani et al. [82], following the relevant standards [80, 81]. In this test, natural climate changes were simulated under accelerated conditions. First, the alkyd film on the glass plate was kept in distilled water at room temperature (25 °C) for 18 h. Then, it was kept in a deep freezer at −20 °C or 3 h. Finally, it was kept in a ventilated oven at +50 °C for 3 h. These three stages were defined as 1 cycle, and 10 cycles were

performed consecutively. Then, the film was examined visually (deformation, detachment from the surface, crack formation, etc.).

3 Results and discussion

3.1 PET depolymerization and characterization of the depolymerization product

The simultaneous hydrolysis-glycolysis reaction of waste PET flakes was carried out using DPG and water at high temperature and pressure in xylene. At the end of the depolymerization reaction, 91.5 g of DP was obtained from 100 g of waste PET loaded into the high-pressure reactor. The depolymerization reaction conditions are presented in Table 1.

3.1.1 End-group analysis of the depolymerization product

The end group amounts of the raw DP obtained at the end of the depolymerization reaction were determined by AV (ASTM D1639-90(1996)e1 standard [66]) and HV (ASTM E222-10 standard [67]) analyses, volumetrically.

The AV of DP was determined by acid-base titration using 0.1 M KOH, and the HV value was determined by acetylation of the sample dissolved in the acetylation reagent prepared with pyridine and acetic anhydride and by the back-titration method with 1 M NaOH.

At the end of these analyses applied to DP, the AV was found to be 341 mg KOH/g, and the HV was 186 mg KOH/g. These results indicate that DP is a mixture of hydroxyl- and carboxyl-terminated oligomers of PET.

Additionally, the theoretical number average molecular mass and equivalent masses based on functionality (E_a and E_b) of DP having hydroxyl and carboxyl end groups were calculated using its AV and HV according to Eq. (1), Eq. (2), and Eq. (3), respectively [83].

$$M_n = \frac{56100}{(\mathrm{AV}+\mathrm{HV})/2} \tag{1}$$

$$E_a = \frac{56100}{\mathrm{AV}} \tag{2}$$

$$E_b = \frac{56100}{\mathrm{HV}} \tag{3}$$

In Eqs. (2) and (3) E_a is the equivalent mass based on carboxyl functional group, E_b is the equivalent mass based on hydroxyl functional group, and 56100 is the molar mass of potassium hydroxide in mg unit.

The characteristics of the DP are presented in Table 2.

As can be seen from these results, at the end of the depolymerization achieved by the simultaneous hydrolysis-glycolysis reaction under high pressure and temperature using water and DPG in the presence of xylene, the PET chain was broken down, and a functional oligomer mixture was obtained having carboxyl and hydroxyl end groups with lower molecular mass.

3.1.2 DSC analysis of the depolymerization product

DSC analysis of DP was performed on a Hitachi Nexta DSC200 (Japan) instrument. In the measurements, 10 mg of sample weighed into an alumina crucible was heated in nitrogen atmosphere from −80 to 400 °C at a rate of 10 °C/min. The DSC curve of DP is given in Fig. 2.

As seen in Fig. 2, more than one endotherm signal is noticed in the DSC curve of DP. Here, instead of a sharp and single melting peak, a broad and relatively sharp melting peak and many small shoulder-type endotherms were observed, depending on the molecular mass distribution. Small endotherms in the shoulder type are intertwined with each other. The large and relatively sharp endotherm is also located in a wide temperature range (285–400 °C). These all indicate that DP is a mixture of oligomers rather than a pure product. The coexistence of monomers, dimers, and oligomers, which are likely to be present in the DP composition, prevented the melting of it in a single stage. As a result, the depolymerization of PET through a simultaneous hydrolysis-glycolysis reaction using DPG and water resulted in an oligomer mixture with varying end group distributions.

To characterize the oligomers comprising the DP composition, the temperature of the endotherm signals obtained from the DSC curve of DP were compared with the melting temperatures of PET oligomers presented in

Table 1 Depolymerization reaction conditions of waste PET

PET/DPG molar ratio	Water (mL)	Xylene (mL)	Catalyst (zinc acetate)	Temperature (°C)	Pressure (bar)	Time (h)
1/1	60	250	1 wt.% of PET	220–225	25–26	3

Table 2 The characteristics of DP

Yield (%)	AV (mg KOH/g)	HV (mg KOH/g)	Number average molecular mass (M_n)	Equivalent mass based on functionality	Color in daylight
91.5	341	186	213	E_a: 165 E_b: 302	Matt yellowish-white

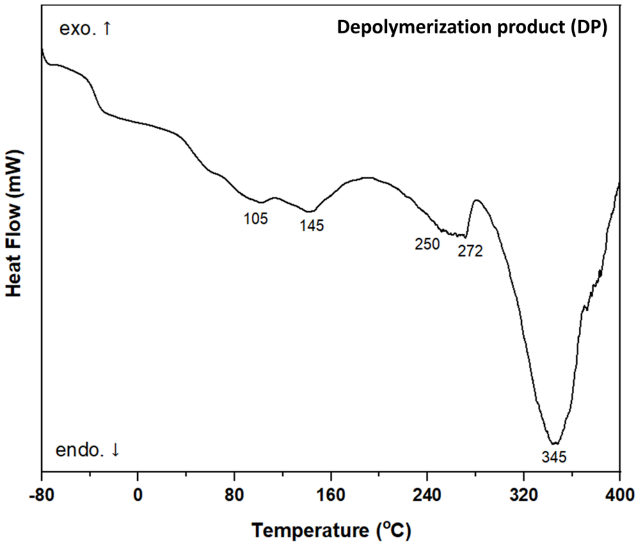


Fig. 2 DSC curve of DP

Brandrup and Immergut [84]. Thus, it was determined which PET oligomer could be attributed to the endotherm signals observed in the DSC curve of DP [85, 86]. The melting temperatures of PET oligomers given in the literature are presented in Table 3 [84].

The slight differences observed in the melting points in the DSC curve of DP, compared to those of PET oligomers reported in the literature, can be attributed to the dissolution in each other and co-melting of monomers, dimers, and oligomers present in the DP composition during the DSC analysis [85, 86]. Accordingly, minor differences or shifts may occur in the temperatures corresponding to the melting points of PET oligomers. When an assessment is made considering this information, the possible oligomer distribution of DP obtained by comparing the melting temperatures of PET oligomers is presented in Table 4.

Table 3 The melting temperatures of PET oligomers [84][*]

Oligomer	*n*	T_m (°C)
$HOOCC_6H_4CO(OCH_2CH_2OOCC_6H_4CO)_nOH$	1	>360
	2	280–281
	3	268–270
	4	252–255
	5	233–236
$H(OCH_2CH_2OOCC_6H_4CO)_nOCH_2CH_2OH$	1	109–110
	2	173–174
	3	200–205
	4	213–216
	5	218–220
$H(OCH_2CH_2OOCC_6H_4CO)_nOH$	1	178
	2	200–205
	3	219–223

[*] In Table 3 *n* is the number of repeated units, T_m is the melting temperature.

Table 4 Possible oligomer distribution of DP

Endotherm signal temperature in DSC (°C)	Possible oligomer equivalent		
	HOOC - ~~~ - COOH	HO - ~~~ - OH	HO - ~~~ - COOH
345	*n* = 1		
272	*n* = 2		
	n = 3		
250	*n* = 4		
	n = 5		
145		*n* = 2	
			n = 1
105		*n* = 1	

As seen in Table 4, DP consists of hydroxyl-terminated monomer (*n* = 1), hydroxyl-terminated dimer (*n* = 2), hydroxyl-carboxyl-terminated monomer (*n* = 1), carboxyl-terminated oligomers (*n* = 2, 3, 4, 5), and mostly carboxyl-terminated monomer (*n* = 1). The formation of carboxyl-terminated oligomers is an expected result in depolymerization reactions occurring in the presence of water [86]. In addition, there is also a possibility that other hydroxyl-, hydroxyl-carboxyl, and carboxyl-terminated oligomers may be present in small amounts in the composition of DP.

As a result, it is seen that DP, which is used as a diol component in the alkyd resin structure, is not a pure product but a mixture of PET oligomers.

3.2 Formulation calculations and syntheses of alkyds

3.2.1 Unmodified alkyds

Firstly, four-component, medium-oil unmodified reference and PET-based alkyd resins (REF-ALK, PET-ALK), in other words, unmodified counterparts of modified resins, were synthesized for comparison with *in situ* aldehyde-modified resins.

REF-ALK without PET was synthesized using TOFA, PA, GLY, and DPG. To synthesize PET-ALK containing PET, DP was used together with other alkyd components, TOFA, PA, and GLY.

Here, the raw simultaneous hydrolysis-glycolysis reaction (depolymerization) product (DP) of PET was incorporated entirely instead of the DPG as a diol component. Thus, DP, a mixture of oligomers with hydroxyl and carboxyl functional groups, also partially replaced the dibasic acid component. In other words, DP completely replaced the diol component (DPG) and partially replaced the dibasic acid component (PA) of the alkyd formulation.

The components of unmodified alkyd resins synthesized at this stage are presented in Table 5.

Table 5 The components of unmodified REF-ALK and PET-ALK alkyds

Components of unmodified resins	Unmodified alkyd resin without PET (REF-ALK)	Unmodified alkyd resin with PET (PET-ALK)
Monobasic acid component	TOFA	TOFA
Dibasic acid component	PA	PA + DP
Polyol component	GLS	GLS
Diol component	DPG	DP

Alkyd resin formulations were prepared using the *K* alkyd constant method [87]. The *K* alkyd constant method is based on an average functionality for the alkyd composition. In this formulation calculation method [87], the *K* constant is the ratio of the total amount of moles (m_o) to the acid equivalent (e_A), and the *R*-value is the ratio of the base equivalent (e_B) to the acid equivalent (e_A).

For REF-ALK, the *K* constant was 1.1, and the *R*-value was 1.15. In the case of PET-ALK, the *K* constant was 1.07, and the *R*-value was 1.15.

Formulation calculations of REF-ALK and PET-ALK alkyds are presented in Table 6.

As mentioned above, DP is a mixture of hydroxyl- and carboxyl-terminated PET oligomers. Hence, it has both (E_a and e_A) and (E_b and e_B) values. Since the DP was used instead of the diol component, when calculating the formulation for PET-ALK, the amount of DP was determined according to its E_b value and base equivalent (e_B). However, since DP contains carboxyl groups in addition to hydroxyl groups, this was also taken into account in the calculations. To determine the final amount of PA, the E_a value of DP was used, and the acid equivalent (e_A) of DP was subtracted from that of the dibasic acid (PA) component in the formulation.

The probable structure of the four-component unmodified reference alkyd resin is proposed in Fig. 3.

3.2.2 *In situ* aldehyde-modified alkyds

In BASF's technical data sheet, it is stated that air and oven-drying alkyd resins are among the fields of application for Laropal A 81, and it is recommended for partial replacement of up to 20% solids in solids and as a modifying component in alkyd resin production [62]. In addition, BASF's industrial coatings raw material selection guide states that it can also be used as a co-binder for improving solids content [88].

Based on this information, chemical modifications of alkyd resins with and without waste PET were conducted *in situ* using aldehyde resin (Laropal A 81) as a modifier.

Table 6 The formulations of unmodified REF-ALK and PET-ALK alkyds[*]

Components of alkyds	Equivalent mass based on functionality (*E*)	Acid equivalent (e_A)	Base equivalent (e_B)	Functional groups number (*F*)	Total number of moles (m_o)
REF-ALK *(K:1.1; R:1.15; U:0.127) (oil-length: 45%)*					
TOFA (monobasic acid)	286	0.1270	–	1	0.1270
PA (dibasic acid)	74	0.3381	–	2	0.1691
GLS (polyol)	31	–	0.3112	3	0.1037
DPG (diol)	67	–	0.2237	2	0.1118
PET-ALK *(K:1.07; R:1.15; U:0.140) (oil-length: 45%)*					
TOFA (monobasic acid)	286	0.1400	–	1	0.1400
PA (dibasic acid)	74	0.3251	–	2	0.1626
GLS (polyol)	31	–	0.4340	3	0.1447
DP (diol + dibasic acid)[**]	E_a: 165 E_b: 302	0.1850	0.1009	2	0.0505

[*] The formulation calculations were made according to the "*K* alkyd constant method" [87]. For DP containing both carboxyl and hydroxyl groups, E_a was calculated based on the carboxyl functional group, and E_b was calculated based on the hydroxyl functional group. e_A and e_B are calculated using the *K*, *R*, and *U* values determined for alkyd synthesis, based on the desired oil-length, in four-component alkyd resin formulation calculations. The optimal *U* value is specified by testing different numerical values to obtain suitable *K* and *R* values based on the acid and base equivalents of the components. *K* and *R* are calculated as follows: $K = m_o/e_A$ and $R = e_B/e_A$. The mathematical formulae used for the formulation calculations of alkyds synthesized by the fatty acid method are not provided here. The formulation calculations for four-component alkyd resins are available in detail in Patton [87].

[**] Equivalent masses (E_a, E_b) of DP were calculated using AV and HV (341 and 186 mg KOH/g) (see Table 2). DP was primarily used entirely instead of the diol component due to its hydroxyl group content, so the *E* value (as E_b) was calculated according to the HV for the DP amount. Due to its carboxyl group content, DP also partially replaced the dibasic acid component. For this reason, its AV, hence E_a, was also used to calculate the e_A of DP, which was then subtracted from that of the dibasic acid component for the PA amount.

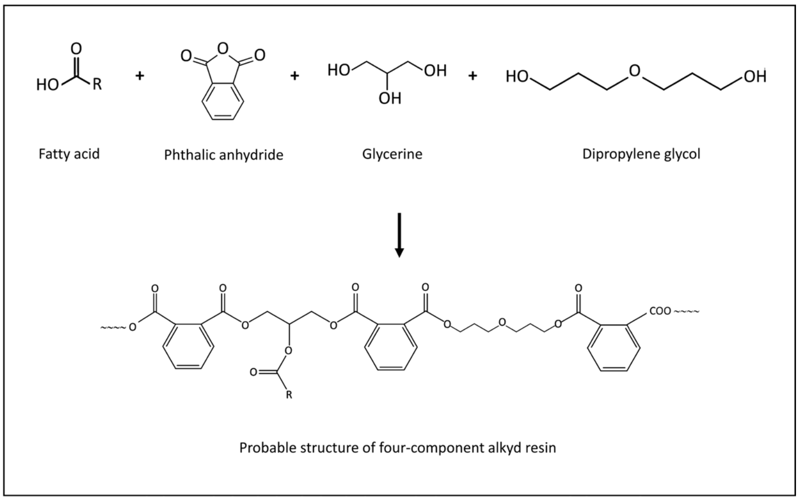


Fig. 3 The probable structure of the four-component unmodified reference alkyd resin

That is, the modification reactions were performed in the reaction mixture during alkyd syntheses.

Formulations of the four-component, medium-oil modified alkyd resins were prepared using the *K* alkyd constant method [87]. The same formulations calculated for unmodified alkyd resins were also used to synthesize modified alkyd resins. For REF-IS-ALK, the *K* constant was 1.1; the *R*-value was 1.15. In the case of PET-IS-ALK, the *K* constant was 1.07; the *R*-value was 1.15.

Similar to unmodified PET-based alkyd resin calculations, DP as a diol component was added to the modified PET-based alkyd resin formulation to substitute the DPG completely. The acid equivalent of DP having both hydroxyl and carboxyl content was subtracted from the acid equivalent of the dibasic acid component (PA) in the formulation calculations.

In both modified alkyd syntheses, the modifier aldehyde resin (Laropal A 81), diluted with 50% xylene by mass, was slowly added to the reaction medium in the last stage (in the last quarter) of the reaction time during the synthesis reaction of this formulation.

Here, based on the information found in the technical data sheet [62] and prior literature [61], it was preferable to use an aldehyde resin at a mass ratio of 7% relative to the alkyd. This ratio aligns with the recommendation that the aldehyde resin should comprise less than 20% of the alkyd's total mass. It is also possible to vary these ratios according to expectations and results achieved.

At the end of the modification reactions, "*in situ* aldehyde-modified reference alkyd resin (REF-IS-ALK)" and "*in situ* aldehyde-modified PET-based alkyd resin (PET-IS-ALK)" were prepared. The components of modified REF-IS-ALK and PET-IS-ALK alkyds and their percentage ratios are presented in Table 7.

3.3 Characterizations of unmodified and modified alkyd resins

3.3.1 FTIR analysis of alkyd resins

FTIR analyses of alkyds were performed on an Agilent Cary 630 (USA) FTIR device. The infrared spectra of samples were obtained in the range of 500–4500 1/cm. The samples were diluted with IR-grade KBr (samples/KBr: 1/200 (w/w)) to prepare the measurement pellets.

FTIR spectra of alkyds are given in Fig. 4. In addition, IR frequencies of the characteristic peaks and corresponding groups in the alkyd resin [58, 59] are also presented in Table 8.

When the FTIR spectra (Fig. 4) of alkyd resins are examined, it is seen that the characteristic peaks belonging to alkyd resin/ester structures shown in Table 8 are

Table 7 The components of modified REF-IS-ALK and PET-IS-ALK alkyds and their percentage ratios

Components of modified resins	Modified alkyd resin without PET (REF-IS-ALK)	Modified alkyd resin with PET (PET-IS-ALK)
TOFA (monobasic acid)	42 wt.%	43 wt.%
PA (dibasic acid)	29 wt.%	11 wt.%
GLS (polyol)	11 wt.%	14 wt.%
DPG (diol)	18 wt.%	–
DP (diol+dibasic acid)	–	32 wt.%
Laropal A 81 (aldehyde resin, modifier)	7 wt.% of alkyd	7 wt.% of alkyd

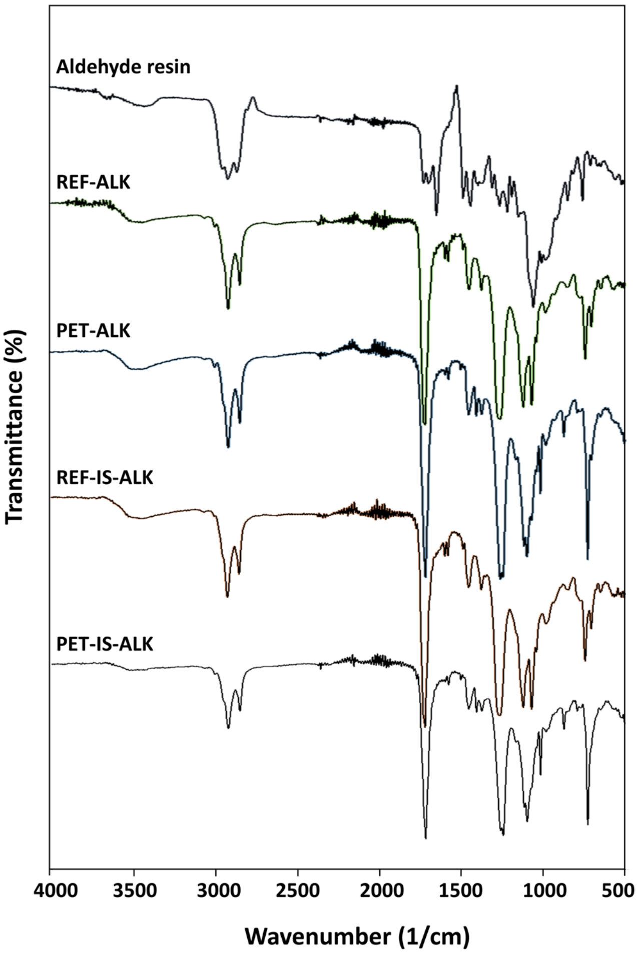


Fig. 4 FTIR spectra of alkyds

Table 8 IR frequencies of peaks and corresponding groups in the alkyds

Group	Frequency (1/cm)
OH (stretching vibration)	3500
C–H (asymmetric and symmetric stretching vibrations)	2925–2850
C=O (stretching vibration)	1730–1720
C=C (aromatic vibration)	1600–1575
C–H (bending vibration)	1450–1380
C–O–C (stretching vibration)	1260–1250
C–O (stretching) (ester)	1120
C–H (in-plane deformation) (aromatic ring)	1070–1060
C–H (out-of-plane bending) (ester)	985–865
C–H (bending and rocking deformation vibrations) (aromatic ring)	785–630

observed in all synthesized alkyds [58, 59]. This finding, as demonstrated by the FTIR spectra of all synthesized products, confirms that the alkyd structure is formed for both the reference resins (REF-ALK and REF-IS-ALK) and the waste PET-based resins (PET-ALK and PET-IS-ALK).

As presented in the literature [59, 63, 64], the IR frequencies of the characteristic peaks and corresponding groups in aldehyde resin can be summarized in the following paragraphs.

Stretching vibration of OH groups at the 3625–3330 1/cm region, stretching vibration of C=O bond in ester and aldehyde structures at the 1730–1710 1/cm region, and stretching vibration of C–N bond in amide groups around 1310 1/cm, stretching vibration of the C=O group in tertiary amide structure at 1640 1/cm.

Nevertheless, the expected differences in the FTIR spectra of aldehyde-modified alkyds are not apparent due to the very low amount of aldehyde resin used compared to alkyd resin, as well as the possible overlapping of the peaks of alkyd and aldehyde resins.

In our previous study on alkyd-aldehyde blends [59], the FTIR spectrum of the alkyd-aldehyde blend containing 20 wt.% aldehyde resin showed that most of the characteristic peaks belonging to aldehyde and alkyd resins overlapped or interfered with each other, and only minor differences were observed in the spectrum. The slight differences observed in the spectrum of the aldehyde-alkyd blend in our previous study [59] not observed in the spectrum of the aldehyde-modified alkyd resin here, probably due to the low amount of aldehyde resin (7 wt.%).

As can be seen in Fig. 4, the spectra of unmodified and aldehyde-modified alkyd resins are quite similar to each other. Considering the possible shifts in the wave numbers of the existing peaks, this situation indicates that almost all of the characteristic peaks attributed to the aldehyde resin [59, 63, 64] in the spectra of modified alkyd resins are not visible because they overlap or interfere with the distinctive peaks of alkyd resin [58, 59]. The possible overlaps and interferences observed in the spectra are summarized in the following paragraphs.

The splay peak belonging to the OH group, which is present in both the alkyd and aldehyde resin structures and is located around 3500 1/cm, is observed in all spectra. It is thought that the 1640 1/cm tertiary amide C=O stretching vibration peak in the aldehyde resin structure is slightly shifted and interfered with the 1730 and 1710 1/cm ester and aldehyde C=O stretching vibration peaks located right next to it, as well as the broad ester C=O stretching vibration peak located in the 1730–1720 1/cm region in the alkyd resin spectrum. It is also possible that the intensity of this peak belonging to the aldehyde resin is low due to the addition of a minimal ratio to the alkyd resin formulation. However, it is believed that the small peak observed around this wave number in all alkyd resin spectra, both modified and unmodified, overlaps with the small aromatic

C=C vibration peak in the 1575–1600 1/cm region in the alkyd resin spectrum. It is considered that the 1311 1/cm amide group C–N stretching vibration peak in the aldehyde resin structure interferes with the broad C–O–C stretching vibration peak located in the 1260–1250 1/cm region in the alkyd resin spectrum.

The overlaps and interferences in the FTIR spectra make it challenging to understand that a possible reaction has occurred. However, differences in DTA, DSC, and TGA analyses, viscosity measurements, and surface coating test results suggest that the expected modification may have taken place.

Although the expected differences in the FTIR spectra were not clearly observed due to the low amount of aldehyde resin, it can be predicted that potential reactions between alkyd and aldehyde would probably occur. Our previous publication [59] suggested that expected small changes could be due to possible reactions between the free hydroxyl (OH) and carboxyl (COOH) groups of the alkyd resin and the carbonyl (C=O) groups in the tertiary amide structure of the aldehyde resin during drying/curing. Additionally, weak interactions between the OH, COOH, and C=O groups may also be considered as contributing factors.

3.3.2 DTA and DSC analyses of alkyd resins

DTA provides information about the thermal properties of the sample, similar to DSC analysis. As is known, DTA and DSC measure the heat input required to increase the temperature of a sample. Both methods compare the thermal properties of a sample and a reference material. In DSC, the differential heat input required to increase the temperature of both the sample and the reference is measured. In DTA, the differential temperature change between the sample and the reference is measured for a fixed amount of heat input [89]. In summary, DSC and DTA are both thermal analysis techniques that use similar measurement principles, providing insight into the phase transitions of a material using a controlled temperature program. DSC measures the difference in heat flow required to keep the sample and the reference material at the same temperature, while DTA measures the temperature difference between the sample and the reference when the applied heat is the same [89, 90].

DTA analyses of alkyds were performed on a Linseis STA PT 1750 (Germany) DTA/TGA combined device. The measurements were realized by heating ~10 mg of the sample from ambient temperature (20–25 °C) to 800 °C at a rate of 10 °C/min in an air atmosphere. The DTA graphs of alkyds are given in Fig. 5.

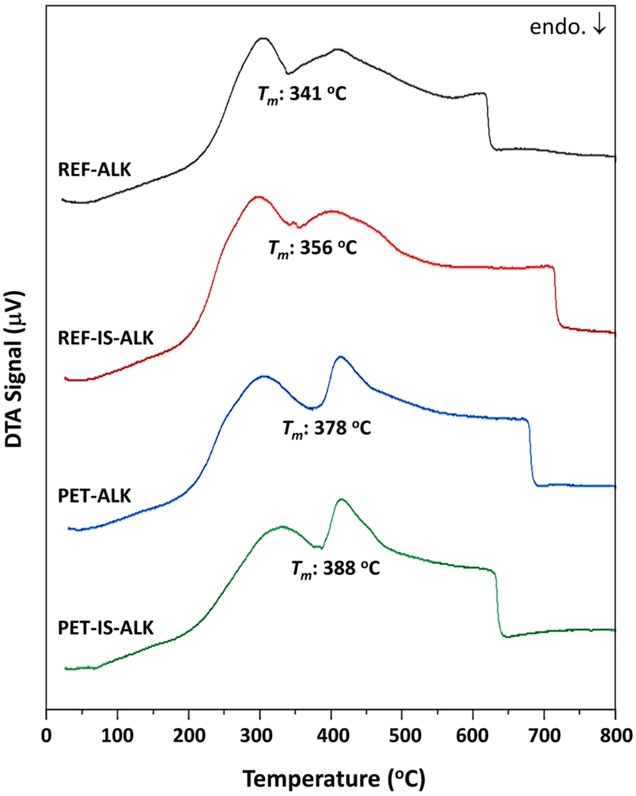


Fig. 5 DTA graphs of alkyds

As seen in the DTA graphs of alkyds, the melting temperatures (T_m values) of REF-ALK, REF-IS-ALK, PET-ALK, and PET-IS-ALK alkyds were found to be 341, 356, 378, and 388 °C, respectively. These values are compatible with the melting temperatures of alkyd resins given in Erol et al. [59], and Islam et al. [91]. In addition, the melting temperatures of aldehyde-modified alkyds are higher than those of their unmodified counterparts, in both reference and PET-based alkyd series.

The fact that modified alkyds' melting temperatures differ significantly from those of their unmodified counterparts probably indicates a structural change and inclusion of aldehyde resin in the alkyd structure.

To observe the thermal behavior in the 25–300°C range in more detail, DSC analyses of alkyd samples were also conducted using a faster scan under nitrogen. DSC analyses of alkyds were performed on a Hitachi Nexta DSC200 (Japan) instrument. Measurements were made by heating ~10 mg samples from room temperature (~25 °C) to 300 °C at a rate of 20 °C/min under a nitrogen atmosphere. DSC curves (second heating) of alkyds are presented in Fig. 6.

In the DSC curves of all alkyd resins in Fig. 6, a broad exotherm is observed at temperatures above 100 °C, likely due to an exothermic reaction. This exotherm is probably related to the curing reaction [92, 93]. Due to possible

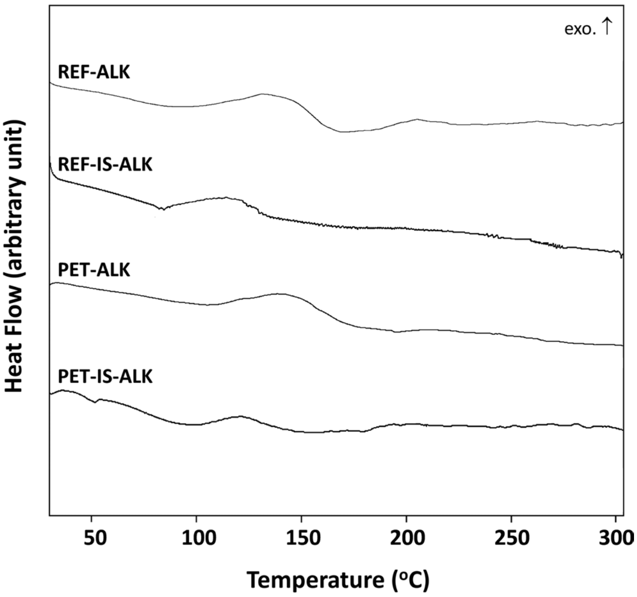


Fig. 6 DSC curves of alkyds

crosslinking reactions at high temperatures, a certain amount of curing may have occurred during the gradual heating process in the analysis. Similar exothermic peaks have also been attributed in the literature to the heat developed during various crosslinking reactions [92].

However, glass transition temperatures (T_g) could not be observed in these curves, possibly because they were below the measurement scale. Typically, T_g is monitored with a slight, gradual change over a wide temperature range, and it never appears as an endothermic peak [93, 94]. It has been stated in the literature that this phenomenon indicates a wide distribution in molecular mass in such thermoset materials [93]. Previously reported T_g values for alkyd resins in the literature have been observed in the range of −20 to 30 °C, although they vary depending on the formulation [95–97].

3.3.3 Other characteristics of alkyd resins

Other characteristics (AV, solids content %, viscosity, and appearance/color) of all synthesized alkyds are presented in Table 9.

The AV of alkyds were determined titrimetrically according to the ASTM D1639-90(1996)e1 standard [66]. The percent nonvolatile (solids) content of alkyds was determined according to ASTM D7232-06 standard [98]. Rotational tests were applied to alkyd resins using an Anton Paar MCR 102 rheometer (Austria), and viscosity measurements were carried out with a parallel plate (PP 25) with a diameter of 25 mm at 25 °C. The appearances and colors of the alkyds were evaluated visually in daylight.

Table 9 Other characteristics of alkyds

Alkyd resin	AV (mg KOH/g)	Solids content (%)	Viscosity (mPa s)	Color in daylight
REF-ALK	30	85	11,398	Dark brown
REF-IS-ALK	28	91	46,402	Dark brown
PET-ALK	18	89	47,984	Brown
PET-IS-ALK	21	83	66,109	Brown

As seen in Table 9, the viscosity values of the PET-based alkyds synthesized with DP at a ratio of 100% instead of the DPG as a diol component is higher than that of the reference alkyds synthesized with DPG. This finding was likely due to the incorporation of the higher molecular mass DP (PET oligomer mixture) into the alkyd structure. However, AV and solids content percentages are close to each other.

To evaluate the physical appearance and color differences of alkyds, actual photographs are presented in glass vials in Fig. 7.

Although no measurements are taken with the device for the color scale, it can be predicted that visual evaluations can give a rough idea of the color and appearance of alkyds. Visual assessments of alkyd resin color and appearance in daylight indicate that all alkyds are translucent. However, visual checks based on comparison with charts showing color scales suggest that the use of waste PET DP causes some lightening of the color and, conversely, aldehyde modification causes darkening of the color.

To provide an accurate evaluation of the color tone of the visually examined samples, the possible Gardner color scale (ASTM D1544-04(2018) standard [99]) correspondences may be proposed as 16, 17, 14, and 15, for REF-ALK, REF-IS-ALK, PET-ALK, and PET-IS-ALK, respectively. Similarly, possible ASTM D1500-24 standard [100] color scale correspondences may also be proposed as 5.0, 5.5, 4.0, and 4.5, respectively. These results imply that the use of waste PET product in alkyd synthesis does not

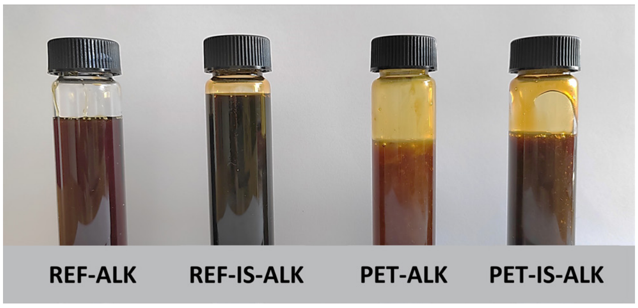


Fig. 7 The photographs of alkyds before and after aldehyde modification

have an adverse effect on color properties; on the contrary, it positively improves this property. Additionally, according to these results, it can also be suggested that the aldehyde modification darkens the color slightly.

3.4 Coating properties of unmodified and modified alkyd resins

3.4.1 Curing of alkyd resin films

First, primary and auxiliary/secondary driers were added to the resins in amounts calculated to contain 0.1% Mn, 0.2% Zr, and 0.39% Pb by mass based on the amount of alkyd, and 50 µm films were prepared with the BYK applicator. Then, the prepared TOFA-based medium-oil unmodified and modified alkyd films were oven-cured.

When the resin films were oven-cured at 140 °C, the modified alkyd films reached the 7th drying stage (full dry) after being cured in the oven for 1 h, but unmodified alkyds required 2 h to achieve this stage. That is, the possibility of aldehyde resin (Laropal A 81) participating in the crosslinking reaction due to its secondary hydroxyl groups might have resulted in higher crosslink density and shorter curing time.

All prepared alkyd films were cured under the same conditions (2 h at 140 °C) after the addition of a drier, to ensure that they reach the 7th drying degree and coating properties can then be determined. Thus, after the oven-curing process, all alkyds reached the full-dry stage. Then, physical and chemical surface coating tests were applied to the oven-cured films.

3.4.2 Physical surface coating test results

The physical surface coating properties of all alkyds were determined according to the relevant standards mentioned above. The coating tests were performed to determine the hardness, adhesion strength, impact resistance, abrasion resistance, and gloss properties of all oven-cured resin films, and the results are presented in Table 10.

As seen in Table 10, relatively hard and glossy films with generally excellent adhesion strength and impact resistance were obtained from alkyds. Detailed explanations are provided below.

Hardness

When the hardness values (Table 10) of the reference (REF-ALK and REF-IS-ALK: 111 and 100 König sec) and waste PET-based alkyd films (PET-ALK and PET-IS-ALK: 129 and 120 König sec) in both unmodified and modified series were compared with each other, it was observed that the hardness values of the PET-based alkyds were slightly higher. Likewise, it has been previously reported in the literature that optimum amounts of waste PET improve the mechanical properties, such as the hardness, of the blends and reaction products prepared from various resins and waste PET [58, 101]. The hardness values of the alkyd resin films containing waste PET product are somewhat higher than those of the counterpart reference resin films, probably due to the waste PET-based oligomeric structure added to the overall structure instead of the diol component. The hydroxyl and carboxyl-terminated PET oligomers may have caused additional crosslinking reactions.

In the case of the hardness values of unmodified (REF-ALK and PET-ALK: 111 and 129 König sec) and modified alkyd films (REF-IS-ALK and PET-IS-ALK: 100 and 120 König sec) in both reference and waste PET-based alkyd series, it was observed that the hardness values of REF-IS-ALK resin and PET-IS-ALK resin films obtained by *in situ* modification reaction with aldehyde resin were slightly lower than their unmodified counterparts (REF-ALK and PET-ALK) at the end of modification. Although there is no significant difference, the difference in hardness values is likely due to the aldehyde resin as a modifying agent added to the reaction medium, and this situation can probably be attributed to the decrease in hardness resulting from the increase in flexibility. The literature has reported that copolymerizing linear glycols with urea and formaldehyde significantly increases the flexibility of cured resins. It has been stated that the glycols in the polymer structure provide flexible soft segments that act as spacers between crosslinking regions [102]. Here, although the crosslinking density increases during the curing process, the modifier aldehyde resin added *in situ* to the medium during the alkyd synthesis may have probably exhibited such an effect together with the diol component present in the reaction medium.

Table 10 The physical surface coating properties of alkyds

Alkyd resin	Pendulum hardness (König sec)	Adhesion strength (%)	Impact resistance (kg × cm)	Abrasion resistance (mL sand)	Gloss (GU)
REF-ALK	111	100	>200	850	147
REF-IS-ALK	100	100	>200	900	131
PET-ALK	129	100	>200	750	108
PET-IS-ALK	120	100	>200	800	115

Adhesion strength

Images of the tested panels for cross-cut adhesion are presented in Fig. 8. The classification table prepared according to ASTM D3359-23 [72] and ISO 2409:2020 [73] standards is also given in Table 11.

At the end of the cross-cut adhesion test of all resin films based on reference and waste PET, unmodified and modified, no peeling/flaking was observed in the lattice pattern created with the 6-blade cross-cutter on the glass plates. As seen in Fig. 8, the affected surface area is 0%. Classification degrees according to ISO 2409:2020 [73] and ASTM D3359-23 [72] standards are 0 and 5B, respectively. That is, their adhesion strength was 100%, and they showed excellent adhesion (Table 10).

According to these results, the use of waste PET product and modification with aldehyde resin did not cause an adverse effect on the adhesion strength, which refers to the strong bond between the coating and the substrate of a substance.

Impact resistance

The impact resistance test results of all alkyd resin films based on unmodified and modified reference and waste PET are greater than 200 kg × cm and show excellent impact resistance (Table 10). The use of waste PET product and modification with aldehyde resin did not adversely affect the impact resistance property, which refers to the ability of a coating to withstand various forces, such as dropping, without breaking or deforming.

Abrasion resistance

The abrasion of coatings depends on various aspects, such as elasticity, hardness, strength, toughness, brittleness, and thickness. That is, the different factors, such as hardness, impact resistance, and flexibility, govern the abrasion resistance of materials [58, 103]. In this context, the abrasion resistance test gives an idea about the impact resistance of the films as well as their flexibility properties and generally (although not always) varies inversely with the hardness of the film [31, 59].

Here, for the abrasion resistance values, a decrease was observed in the order REF-IS-ALK > REF-ALK > PET-IS-ALK > PET-ALK (900 > 850 > 800 > 750 mL sand), and they varied inversely with the hardness values (Table 10). Additionally, it is observed that the abrasion resistance values of the modified alkyd resin films synthesized with the incorporation of aldehyde resin in the *in situ* modification reaction are slightly increased compared to their unmodified counterparts, and somewhat more flexible films are formed.

This case, as also mentioned in the passage about the hardness values of the films, is probably due to the formation of more flexible structures [102] by the interaction of the aldehyde resin added to the medium during the *in situ* modification stage with the diol component in the alkyd synthesis reaction. It is thought that adding long-chain oligomeric waste PET product also contributed positively to this result [26].

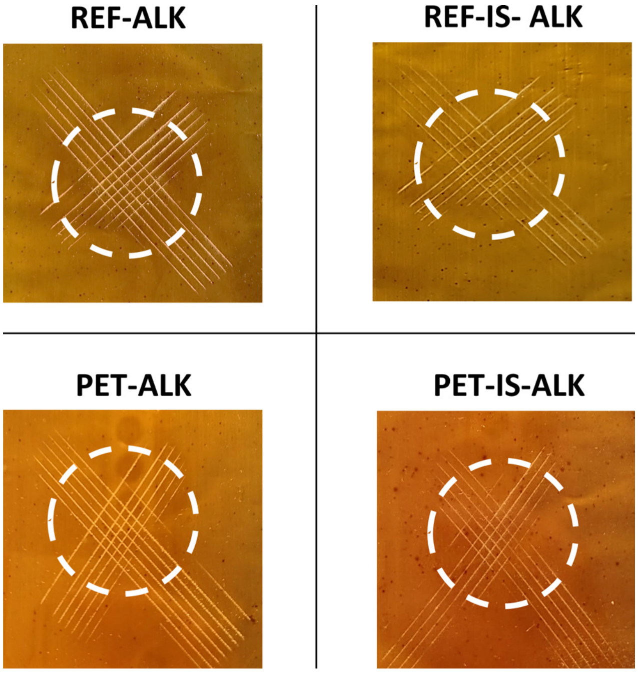
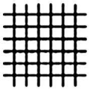
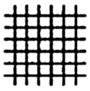
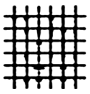
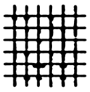
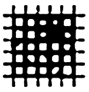
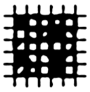
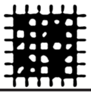
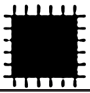


Fig. 8 Images of the tested panels for cross-cut adhesion

Table 11 Classification table for cross-cut adhesion test

Surface appearance of cross-cut area		Definition/explanation	Affected cross-cut area	Classification	
Min. removal	Max. removal			ISO 2409:2020 [73]	ASTM D3359-23 [72]
		*None*: the edges of the cuts are completely smooth, and none of the squares in the lattice are detached.	0%	0	5B
		*Minor*: there is detachment of flakes from the coating at the intersections of the cuts.	< 5%	1	4B
		*Moderate*: the coating has flaked along the edges and/or at the intersections of the cuts.	5–15%	2	3B
		*Significant*: the coating has flaked along the edges of the cuts either partly or entirely in large ribbons, and flaked either partly or entirely in different parts of the squares.	15–35%	3	2B
		*Severe*: the coating has flaked along the edges of the cuts in large ribbons, and some squares be partially or completely detached.	35–65%	4	1B
		*Excessive*: any degree of flaking that cannot be categorized under classification 1B or 4.	> 65%	5	0B

Gloss

The gloss test results are shown in Table 10 for all alkyd films. The gloss values of films in the unmodified alkyd resin series were found to be 147 and 108 GU for REF-ALK and PET-ALK, respectively. Although adding waste PET product to alkyd caused a decrease in gloss, a relatively high gloss film was still obtained. The gloss values of films in the modified alkyd series were observed to be 131 and 115 GU for REF-IS-ALK and PET-IS-ALK, respectively. As can be seen, very glossy alkyd films similar to unmodified counterparts were obtained after *in situ* modification with aldehyde resin, in both series.

In the PET-based alkyd series, as expected, a slight increase (from 108 to 115 GU) was observed on gloss after modification with aldehyde resin, which has a gloss-enhancing effect, while a decrease (from 147 to 131 GU) was observed on gloss in the reference series. This different trend in gloss property observed in the reference and PET-based alkyd series is also probably related to the curing process.

As previously reported in the literature, the curing process is critical for gloss, and the process conditions affect the gloss of alkyds [104–106]. Here again, the oven-curing process is likely to have slightly affected the gloss of the alkyds. In addition, the gloss is also affected by various factors, such as the sample behavior (the defects, such as yellowing) during curing, besides the curing conditions [106, 107]. These defects lower the gloss and reduce the distinctness of the image. This situation is seen more noticeably in high-gloss coatings [108].

Also, as it is known, yellowing is caused by over-oxidation of the coating and is a general problem for alkyd coatings [109]. Yellowing, gloss, and color retention (and/or difference) are closely related to the curing process [106, 110]. The presence of remaining unsaturated bonds in the cured coating causes the coating to turn yellow following oxidation after exposure to atmospheric oxygen [111], which may cause the surface to appear dull (lose some of its gloss) due to color change (discoloration) [112].

In conclusion, when the PET-based and reference alkyd series are compared, it is observed that adding waste PET product to the alkyd formulation reduced (from 147 to 108 GU) the gloss of the PET-based alkyd with respect to the counterpart reference alkyd. In contrast, incorporating aldehyde resin into the PET-based alkyd structure by *in situ* modification slightly increased (from 108 to 115 GU) the gloss of the modified PET-based alkyd. This result shows that Laropal A 81 can eliminate the gloss loss that occurs in alkyd resins containing PET product, as seen in the *in situ* modified PET-IS-ALK.

3.4.3 Chemical surface coating test results

To determine the chemical surface coating properties of all oven-cured alkyd resin films, alkali resistance, acid resistance, salt resistance, water resistance, solvent resistance, and environmental resistance tests were applied according to the relevant standards as mentioned above.

Alkali, acid, salt, water, and environmental resistance test results are presented in Table 12. In addition, solvent resistance (acetone, methanol, toluene, and ethyl acetate) test results are also presented in Table 13.

Alkali resistance

The evaluation of alkali resistance test results (see Table 12) indicated that all resin films are significantly affected by the alkali solution. This situation is expected because alkyd resins generally possess poor alkali resistance due to the presence of hydrolysable ester bonds [113]. However, the reference unmodified alkyd resin resisted the alkali solution for only 45 min, while the waste PET-based unmodified resin resisted the alkali solution for 180 min. Incorporation of long-chain waste PET oligomers into the alkyd structure instead of the diol component slightly enhanced the alkali resistance.

When unmodified alkyds were compared with their modified counterparts, an albeit somewhat positive effect of *in situ* modification with aldehyde resin on alkali resistance was observed, which means that their alkali resistance can be improved by various modifications using the optimum aldehyde resin ratios to be determined.

Acid, salt, and water resistance

Upon examining acid, salt, and water resistance test results (Table 12), it is seen that all alkyd resin films are not affected by corrosive chemicals such as acid, salt, and water under static conditions and for relatively long periods, and their corrosion resistance is high.

Images of alkyd films tested in a salt solution by immersion technique for 120 h are presented in Figs. 9 and 10, both during and after the test. As seen in the images presented in Figs. 9 and 10, there are no signs of corrosion around the X mark drawn on the alkyd films immersed in 5 wt.%. NaCl solution after 120 h (5 days).

Instead of X-cut salt spray tests in a corrosion test chamber to evaluate the corrosion resistance of coatings, chemical resistance tests, such as acid, salt, and water, can provide a practical idea of the coating's corrosion resistance. The literature reports the use of a 3.5 wt.%. NaCl solution in determining the corrosion resistance of coatings [114]. Here, concentrated solutions (3 wt.% sulfuric acid and 5 wt.% sodium chloride) were used in the acid and salt resistance tests, and the films were unaffected by the acid solution even after 72 h (3 days), and by the salt solution even after 120 h (5 days). Additionally, in water resistance tests conducted at room temperature, alkyd films showed no degradation even after 18 h of water immersion, according to the ASTM D1647-89(1996)e1 standard [78]. Considering that the aggressive chemicals used in these tests have an effect on the corrosion resistance of the materials and that the coating materials are designed for daily life applications, it can be concluded that the synthesized modified alkyds have high chemical resistance, indicating good corrosion resistance.

Environmental resistance

Designed as an accelerated cyclical simulation of changing climatic conditions, the repeated changes of hot/cold and wet/dry conditions put a lot of stress on the coating system. Therefore, this test provides an idea about the resistance and durability of the coating to environmental conditions. When the environmental resistance test results are examined (Table 12), it is seen that all alkyd resin films are not affected by cyclic ecological conditions during 10 cycles and show excellent environmental strength.

Table 12 The chemical surface coating properties of alkyds

Alkyd resin	Alkali resistance (0.1 M NaOH) (endurance time)	Acid resistance (3 wt.% H_2SO_4) (for 72 h)	Salt resistance (5 wt.% NaCl) (for 120 h)	Water resistance (distilled water) (for 18 h)	Environmental resistance (for 10 cycle)
REF-ALK	45 min	No change	No change	No change	No change
REF-IS-ALK	60 min	No change	No change	No change	No change
PET-ALK	180 min	No change	No change	No change	No change
PET-IS-ALK	195 min	No change	No change	No change	No change

Table 13 Solvent resistance of alkyds

Alkyd resin	Acetone resistance	Methanol resistance	Toluene resistance	Ethyl acetate resistance
REF-ALK	No change	No change	No change	No change
REF-IS-ALK	No change	No change	No change	No change
PET-ALK	No change	No change	No change	No change
PET-IS-ALK	No change	No change	No change	No change

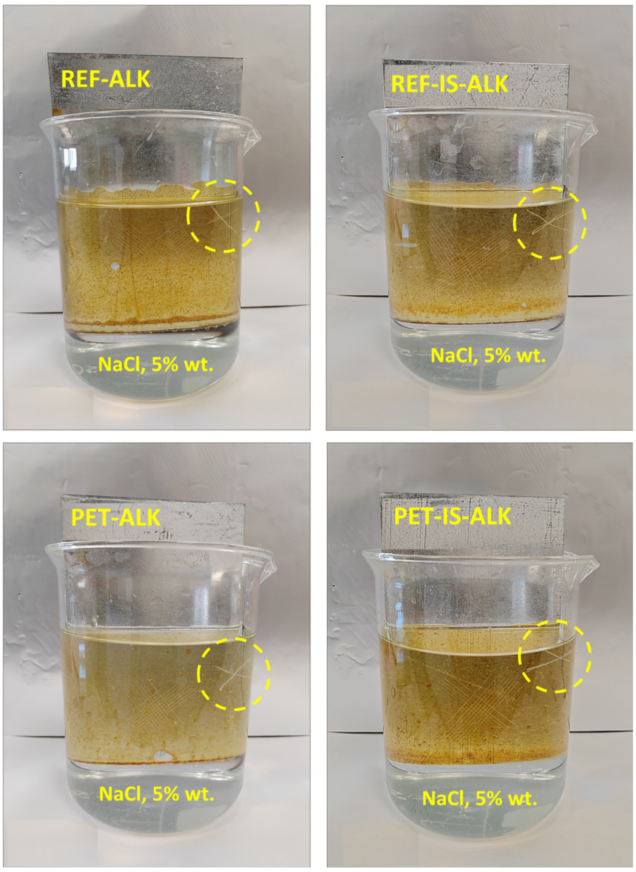


Fig. 9 Images of the panels during salt resistance test

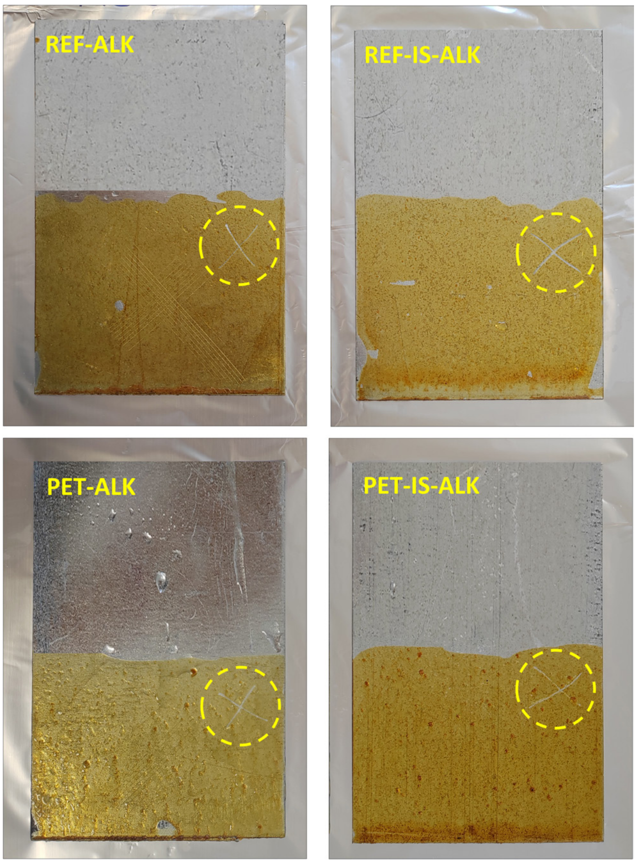


Fig. 10 Images of the panels after salt resistance test

Solvent resistance

The application of the solvent resistance test is presented in Fig. 11.

When the solvent resistance test results shown in Table 13 are examined, it is seen that all resin films based on reference and waste PET, unmodified and modified, are not affected by any aliphatic and aromatic solvents used in this test and show excellent solvent resistance.

As a final result, the use of waste PET product and modification with aldehyde resin did not have an adverse effect on the chemical durability, corrosion resistance, and resistance to the environmental conditions of the resin films.

3.5 Thermal analysis of unmodified and modified alkyd resins

Thermal analysis is an approach in which the properties of a sample are monitored while the temperature in a controlled atmosphere is being varied. As is known, when heat is applied, polymeric materials undergo physical and chemical changes that affect their properties. The relationships between thermal properties and polymer structure can be characterized by various thermal analysis procedures and different methods that can be used to determine the thermal behaviors (or properties) of polymers. In addition to DTA and DSC mentioned earlier, another thermal analysis technique is thermogravimetry, which is typically

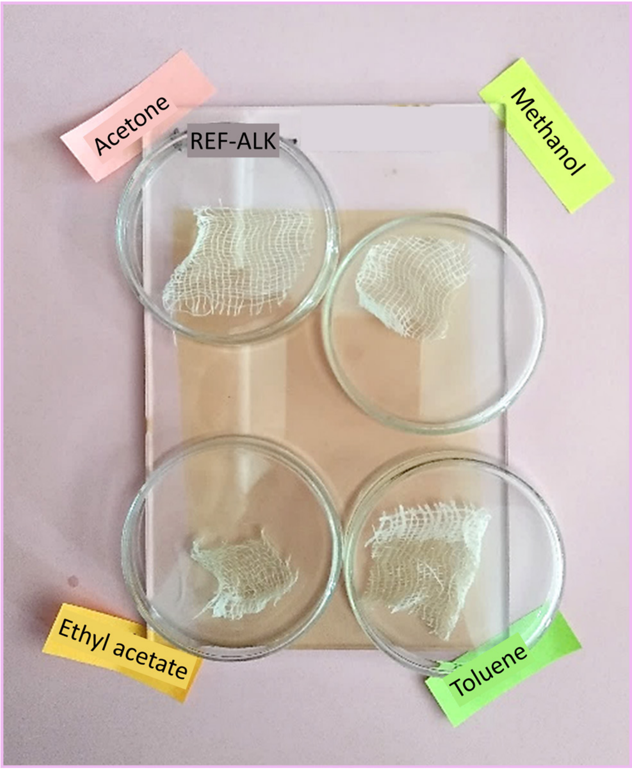


Fig. 11 The application of the solvent resistance test

used to determine mass loss in a material as a function of temperature or time. In TGA, a small amount of sample is heated in an inert or air atmosphere, and any resulting mass changes are measured as a function of temperature. The primary function of TGA is to monitor the thermal stability of a material by recording the change in mass of the sample with temperature. In other words, TGA is a technique used in thermal analysis to measure the change in mass of a sample as it is heated, cooled, or held at a constant temperature in a defined atmosphere. This method allows the characterization of polymers, including their thermal degradation [89, 90, 115].

TGA analyses of unmodified and modified alkyd resin films were performed using a Linseis STA PT 1750 TGA/DTA combined device. Measurements were carried out by heating from room temperature to 800 °C in an air atmosphere at a rate of 10 °C/min.

The TGA graphs of alkyd resin films are given in Fig. 12. The temperature values corresponding to the specific % mass losses of alkyd resins obtained from the TGA curves are also shown in Table 14.

In previous studies, it was stated that the maximum decomposition temperature of alkyd resin was observed at 400 °C, and the mass losses during thermal oxidative decomposition of resin were due to the decomposition of fatty acid chain and polyester backbone [47, 115, 116].

In addition, it has been stated in the literature that the mass loss observed above 400 °C can be attributed to the decomposition of the cross-linked alkyd blend structure containing urea-aldehyde modifier [59].

When the TGA graphs are examined, it is seen that the thermal behaviors of all alkyd films are relatively similar, and heat-stable films with generally high thermal resistance/stability are obtained from alkyds. However, when the thermal behaviors of the unmodified and modified alkyd series are examined separately, the observed minor differences are presented below.

In the unmodified alkyd series, the temperature values observed at the first 10% and 20% mass losses were found to be very close to each other for REF-ALK and PET-ALK. As shown in Table 14, the $T_{10\%}$ values are 209 and 211 °C; the $T_{20\%}$ values are 236 and 239 °C for the unmodified REF-ALK and PET-ALK. This observed phenomenon may suggest that, in practical applications, there is no significant difference in initial thermal stability between unmodified alkyds. However, PET-ALK required a higher

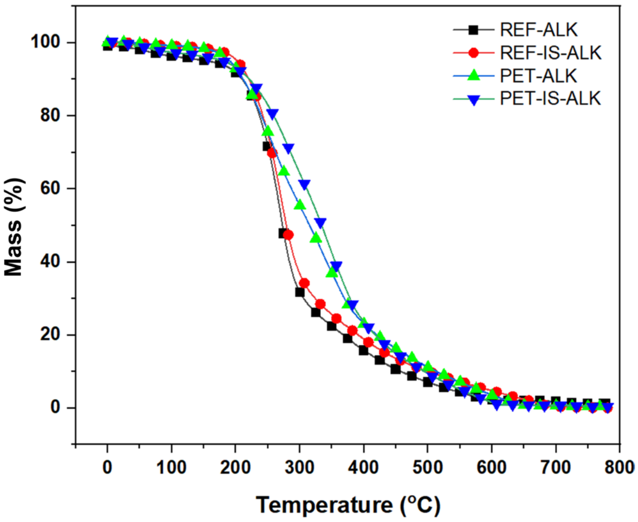


Fig. 12 TGA graphs of alkyd films

Table 14 The temperature values corresponding to the specific % mass losses of the alkyd resins

Alkyd resin	The temperature corresponding to a specific percentage of mass loss									
	$T_{10\%}$	$T_{20\%}$	$T_{30\%}$	$T_{40\%}$	$T_{50\%}$	$T_{60\%}$	$T_{70\%}$	$T_{80\%}$	$T_{90\%}$	T_{Final}^{*}
Unmodified										
REF-ALK	209	236	250	263	272	283	306	366	457	600
PET-ALK	211	239	262	287	315	342	369	416	498	650
Modified										
REF-IS-ALK	220	243	256	268	279	292	323	391	496	670
PET-IS-ALK	221	258	285	311	334	355	378	418	500	680

[*] Final thermal oxidative degradation temperature

temperature than REF-ALK to achieve the same degradation percentage, starting from a 30% mass loss. For example, $T_{30\%}$ values of REF-ALK and PET-ALK are 250 and 262 °C, respectively. Although the initial degradation temperatures ($T_{10\%}$ and $T_{20\%}$) have not changed much, differences in temperatures corresponding to relatively low mass losses, such as 30%, can also give us an idea about thermal resistance/stability. In this context, the degradation temperatures at 30% mass loss ($T_{30\%}$) seen in the TGA graphs may mean that the PET-based alkyd provides higher thermal stability.

Moreover, considering the PET content, when a comparison is made regarding the mass losses observed at advanced temperatures for both unmodified and modified alkyd series, it is seen that the thermal resistance increases in the order of REF-ALK < REF-IS-ALK < PET-ALK < PET-IS-ALK for mass losses of 30% and above, and this trend continues with small fluctuations up to 90% mass loss. This case is likely due to the use of a 100% ratio of waste PET product instead of the diol component in the alkyd resin synthesis. Likewise, the literature has previously declared that blends and reaction products prepared from phenolic resin and waste PET improve stability and prevent degradation up to a specific temperature [117]. In addition, the increase in thermal stability of alkyd resins containing PET intermediates has been attributed to the presence of oligomeric intermediates, which are bigger aromatic molecules in the alkyd structure [83, 118]. Therefore, here, the thermal resistances of PET-based alkyd films were observed to be higher than those of their PET-free reference counterparts in both series, due to the use of long-chain PET oligomer with an aromatic unit.

In the case of aldehyde-modified alkyd series, for REF-IS-ALK and PET-IS-ALK films, $T_{10\%}$ values (220 and 221 °C) were observed close to each other with approximately 10 °C increase compared to unmodified counterpart REF-ALK and PET-ALK films (209 and 211 °C). Although the $T_{10\%}$ values of the modified alkyd films are close to each other and there is no significant difference in thermal resistance between these alkyd films at the start, the observation of higher $T_{10\%}$ values compared to the unmodified counterparts indicates the positive contribution of the modification to the thermal resistance.

Moreover, in the modified alkyds, there is a 15 °C difference between their temperatures corresponding to 20% mass loss of REF-IS-ALK and PET-IS-ALK (243 and 258 °C). This result means that the thermal stability of modified alkyds starts at a lower mass loss value (20%) than that of the unmodified alkyds. As mentioned above, in the unmodified alkyds, the thermal resistance difference between the reference and PET-based alkyds starts to be observed from 30% mass loss. In contrast, in the modified alkyds, this is observed from 20% mass loss, probably due to the effect of aldehyde modification.

In addition, the positive contribution of aldehyde resin to thermal resistance is also clearly observed from all temperature values corresponding to the mass losses of unmodified and *in situ* modified resins (Table 14). Here, the modifier (aldehyde resin) in the modified alkyd structure may react with additional cross-linking and contribute to the cross-linked network [59].

Consequently, the high thermal stability observed in alkyds is probably related to the joint effect of the possible additional cross-linking reactions of both the modifying hydroxyl-terminated aldehyde resin and the hydroxyl- and carboxyl-terminated PET oligomer mixture used instead of the diol.

4 Conclusions

Waste PET product (DP) was successfully incorporated into the alkyd resin formulation, entirely replacing DPG as a diol component and partially substituting PA as a diacid component. Both the waste PET product used as a raw material and the aldehyde resin (Laropal A 81) used as a modifier were compatible with other alkyd components, and alkyd synthesis reactions were carried out without any problems.

At the end of the modification process using the *in situ* reaction method, alkyd films were obtained that are generally hard and very glossy, and possess superior adhesion strength, impact resistance, and high thermal resistance. These films also exhibited excellent resistance to acid, salt, water, and solvents. They remained unaffected by environmental conditions.

The physical and chemical surface coating properties of both modified and unmodified resins were found to be sufficient for various coating applications. This indicates that aldehyde resin is compatible with the alkyd resin structure and does not introduce any adverse effects during their interaction. Additionally, the incorporation of waste PET product in the synthesis of modified and unmodified alkyd resins did not negatively impact the physical/chemical surface coating properties and thermal properties of the films. In fact, similar and/or superior performance was achieved with respect to the reference resins that did not contain PET.

The addition of waste PET product somewhat affected the hardness, alkali resistance, and thermal stability.

In both modified PET-based and modified reference alkyds, a positive contribution to their thermal resistance was observed compared to their unmodified counterparts, likely due to additional cross-linking reactions involving the hydroxyl groups of the aldehyde. Also, incorporating waste PET product into the alkyd structure improved the thermal stability to a certain extent, likely due to the presence of aromatic molecules.

Overall, both unmodified and modified alkyds, regardless of PET content, produced highly glossy films. Although the use of waste PET product slightly reduced gloss, the addition of the aldehyde resin as a modifier counteracted this effect and increased the gloss level. This finding demonstrates that modifying waste PET-based alkyd resins with aldehyde resin can effectively maintain gloss.

In conclusion, this study successfully developed durable, highly glossy, physically and chemically resistant, and heat-stable cross-linked waste PET-based in situ modified aldehyde-alkyd systems that are suitable for versatile coating applications, with a focus on a sustainable approach through the utilization of waste PET bottles.

Acknowledgements

This study is a part of the master's thesis titled "Modification of waste PET-based alkyd resins with aldehyde and ketone resins" prepared at Istanbul University-Cerrahpaşa, Institute of Graduate Studies. The authors thank İlhan Kurt, MSc, for his assistance in providing the aldehyde resin, and Eren Yıldırım, PhD, for his support with the thermal analyses. The authors also thank İrfan Acar, PhD, for his assistance in improving the English quality of the article.

This study was funded by the Scientific Research Projects Coordination Unit of Istanbul University-Cerrahpaşa (IUC-BAP). Project number: FYL-2019-32727.

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