Research Article

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Synthesis of low-VOC unsaturated polyester coatings for electrical insulation

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Abstract: The objective of this research is to develop an unsaturated polyester (UPE) varnish with low-volatile organic compounds (VOCs). Instead of using a solvent, the solvent-free varnish incorporates a reactive diluent to reduce viscosity and a catalyst to accelerate curing. To achieve this, vinyl toluene and 1,4-butanediol dimethacrylate were employed as curing agents. Sebacic acid (SA) and fumaric acid were utilized to create UPE coatings for electrical insulation. Various tests and measurements were made to evaluate the physical, thermal, and chemical structure determination, and electrical properties of the synthesized resins. Given the increasing demand for eco-friendly and low-VOC products, gas chromatography was employed to determine VOC levels. The study demonstrated that the electrical volume resistance of cross-linked coatings containing FA was 1.58×10^{15} Ω·cm, whereas coatings containing SA exhibited a measurement of 6.96×10^{11} Ω·cm. VOC levels in the UPE coatings were found to be in the range of 2.10–3.60%.

Keywords: electrical insulation, UPE, coatings, low VOC, insulating varnish

1 Introduction

The coating insulation system, though regarded as a non-torque-generating and passive component, plays a significant role in places where electricity is utilized. The primary function of this coating is to serve as a fundamental thermal and electrical resistance element, greatly influencing the cooling and heating dynamics of the machinery (1). The approach employed to achieve this objective involves the creation of solvent-free resin systems, specifically focusing on unsaturated polyester (UPE) resins. In the context of polyester resins, the level of volatile organic compounds (VOC) can be primarily attributed to the presence of the toxic cross-linking monomer within the resin. This cross-linking monomer serves a dual role in the polymerization process: it functions as a solvent, reducing the viscosity of the base resin, while also actively participating in the curing process (2). Various polymeric structures, including polyesterimide (2), alkyd (3), epoxy (4), polyurethane (5), and UPE (6), have been synthesized as electrical insulation varnishes.

UPEs, known for their advantageous traits such as low VOC content, excellent dielectric insulation, thermal properties, and cost-effectiveness (7–9), are the preferred choice. UPE is a linear polymer formed through a polycondensation process, combining ester bonds and unsaturated double bonds, typically involving unsaturated dibasic acids with diols or saturated dibasic acids with unsaturated diols (10,11). Common starting materials include maleic anhydride (MA), phthalic anhydride, and propylene glycol for UPE production. Anhydrides are favored over diacids due to their faster esterification rates and lower water content requirements during polycondensation. Glycols are also added to enhance UPE chain flexibility. There exists a wide range of formulations for UPE, utilizing various combinations to optimize the properties of the cured resin (12,13). The curing process for UPE relies on radical polymerization, employing different techniques to initiate free radical formation, such as heat, light, electron beams, and ultrasonic waves. However, practical applications typically involve the use of free radical initiators (10).

UPEs are cured by initiating cross-linking among polymer chains with vinyl monomers like styrene, diallyl phthalate, methyl methacrylate, vinyl toluene (VT), divinyl benzene, or their combinations (14,15). While UPE resins combined with styrene are extensively used in coating technology, concerns over volatility and safety limit their use. In coating formulations, polyester resin is typically dissolved in the vinyl

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monomer, followed by cross-linking and curing facilitated by catalyst-induced stirring (16,17). To address these concerns, 1,4-butanediol dimethacrylate (BDDMA) emerges as a promising alternative. This is due to its compatibility with polyesters, low viscosity, and minimal volatility during polymerization (1–3%) (18). Jiang et al. conducted research on hydroxyethyl acrylate-blocking UPE solvent-less impregnating varnish, which demonstrated an electrical volume resistivity of 1.4×10^{14} Ω·cm, with a volatile content of 4.6%. In the study, it was emphasized that the impregnation varnish has excellent electrical insulation properties with low VOC (19). In a separate study by Sharma et al., the focus was on investigating the influence of nano/micro silica on the electrical properties of UPE composites. When 3% nano silica was added, the electrical volume resistivity was notably enhanced to 25×10^{15} Ω·cm. They found that the electrical characteristics were enhanced by the inclusion of nano-silica as compared with micro-silica (20).

In this research, UPE coatings are produced, which are tailored for electrical insulation using two distinct monomers: Sebacic acid (SA) and fumaric acid (FA). These UPE coatings underwent curing processes employing two diverse reactive monomers: VT and BDDMA. The primary goal of this study was to determine the combination of reactive monomer and acid monomer that would result in superior electrical and thermal insulation properties. As a result, the findings from this research will provide valuable insights for future endeavors in insulating varnish development by the authors. This investigation explored the influence of various monomer characteristics on UPE attributes, and to the best of the author's knowledge, no similar study has been documented in the existing literature. To assess resin qualities, multiple physical tests and electrical measurements were conducted. Chemical structural analysis of the resin was carried out using Fourier transform infrared (FTIR) spectrophotometry. Thermal stability of the cured samples was evaluated using a Thermogravimetric Analyzer (TGA). Furthermore, in line with the demand for environmentally friendly products with very low VOC emissions, VOC tests were conducted using gas chromatography (GC).

2 Materials and methods

2.1 Materials

FA (commercial-grade), SA (commercial-grade), tetrahydrophthalic anhydride (THPA) (analytical-reagent-grade), MA (commercial-grade), neopentyl glycol (NPG) (commercial-grade), butylstannoic acid (catalyst) (analytical-reagent-grade), toluene (commercial-grade), hydroquinone (analytical-reagent-grade), VT (analytical-reagent-grade), and BDDMA (commercial-grade) were used.

2.2 Preparation of UPE varnish

Figure 1 shows that the experimental reaction system used for synthesizing UPE consists of a temperature-controlled glass polymerization reactor with a reflux condenser, mechanical stirrer, combined heating and cooling unit, nitrogen gas inlet, and outlet. Dean–Stark apparatus separates the water from the reaction by distillation.

In the formulation of the UPE resin, a ratio of 1.3:1 was deliberately chosen for the unsaturated component to the saturated component, representing the ratio of total −OH groups to total −COOH groups. The precise distribution of these compounds is outlined comprehensively in Table 1. The process commences with the initial combination of the required quantities of glycol and acid. Esterification is meticulously conducted under controlled conditions, utilizing a suitable catalyst, while maintaining the reaction temperature within the range of 160–170°C. To eliminate the water produced as a by-product during the reaction,

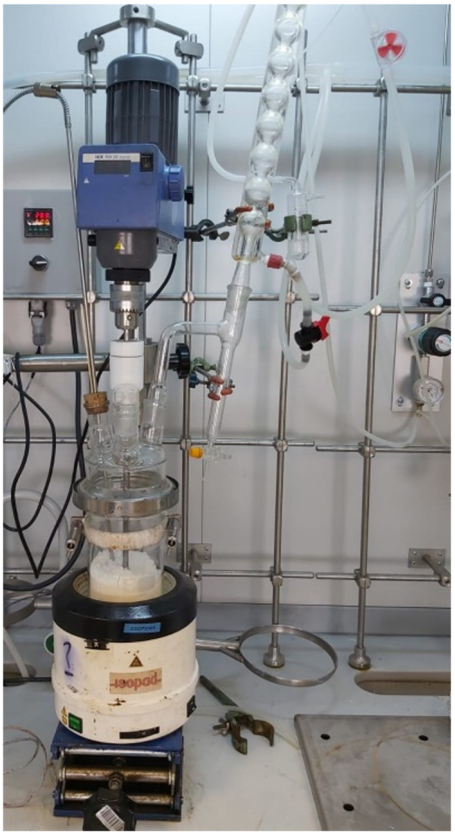


Figure 1: The experimental system of the reaction.

Table 1: Formulation of UPE resin

	UPE-FA-VT	UPE-FA-BDDMA	UPE-SA-VT	UPE-SA-BDDMA
FA	14.84 (0.5)	15.10 (0.5)	—	—
SA	–	–	27.14 (0.5)	27.24 (0.5)
NPG	34.70 (1.3)	35.14 (1.3)	36.20 (1.3)	36.50 (1.3)
THPA	9.75 (0.25)	9.90 (0.25)	10.20 (0.25)	10.30 (0.25)
MA	6.30 (0.25)	6.40 (0.25)	6.55 (0.25)	6.60 (0.25)
VT	30.30 (1.00)	—	15.80 (0.50)	—
BDDMA	—	29.35 (0.50)	—	15.25 (0.25)
Catalyst	0.10	0.10	0.10	0.10
Hydroquinone	0.01	0.01	0.01	0.01
Toluene	4.00	4.00	4.00	4.00
Total (%w)	100.00	100.00	100.00	100.00

Note: The molar ratio of the compounds is shown in parentheses.

azeotropic distillation is employed. Toluene serves as the distillation solvent for the purpose of removing water from the reaction mixture. The progression of the reaction is closely monitored by tracking the acid number in accordance with the ASTM D1639 standard test method. When the acid value of the reaction attains the range of 40–50 mg KOH g^{-1} sample, the reactor is promptly cooled to a temperature ranging from 100°C to 120°C. Subsequently, anhydrides are introduced, and the reaction temperature is diligently maintained between 170°C and 190°C, with continuous removal of the side product, water. Upon reaching an acid value within the range of 10–20 mg KOH g^{-1} sample, the reaction is halted, and hydroquinone is employed to quench it, followed by cooling to room temperature. Figure 2 provides an illustration of a conceivable esterification reaction mechanism.

2.3 Sample preparation

During the curing process, a curing catalyst, comprising 1–3% of the total weight, is introduced into the synthesized resin. Subsequently, the mixture undergoes agitation at 1,000 rpm for a duration of 1 h. In their uncured state, all liquid samples are then subjected to a comprehensive set of tests, including assessments of viscosity, density, VOC content, FTIR analysis, and flash point measurements. To thermal and electrical characterization, the samples are cast into glass Petri dishes and subsequently subjected to curing at a temperature of 200°C for a period ranging from 3 to 5 h. Notably, the curing duration for the VT-included samples is 5 h, whereas the BDDMA samples require 3 h to achieve complete curing. UPE samples synthesized in the study are coded as follows: UPE-SA-VT/UPE-SA-BDDMA and UPE-FA-VT/UPE-FA-BDDMA. The material at the end of the coding indicates the reactive monomer type.

2.4 Characterization

The characterization of the synthesized UPE samples involved several testing procedures.

Viscosity measurements were conducted using a Brookfield viscometer (ASTM D2196) in the uncured liquid state of UPE samples. Various speeds and spindles were employed for this purpose.

The density of the synthesized samples was determined by utilizing a pycnometer at a temperature of 25°C, following the guidelines outlined in the ASTM D1475 standard.

VOC content was assessed through GC in accordance with the ISO 11890-2 standard. Measurements were conducted using a SHIMADZU GC-2030 model GC device with an Rtx-624 column (30 m, 0.25 mm ID, Crossbond 6% cyanopropylphenyl/94% dimethyl polysiloxane) and a temperature range of 50–220°C.

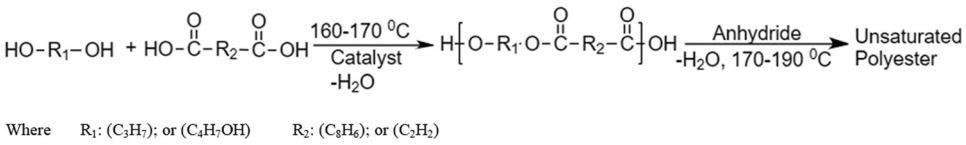


Figure 2: Esterification reaction mechanism.

To determine the percentage solid content, a 1.5–1.6 g sample was placed in an aluminum pan and subjected to an oven at 135°C for a duration of 3 h, following the ASTM D115 procedure.

Flash point measurements were carried out using the Abel closed cup method, adhering to the ISO 13736 standard.

To assess the chemical structure of the cured samples, FTIR spectrometry with an Attenuated total reflectance apparatus was utilized. This analysis was conducted using a Shimadzu Corp. Iraffinity-1S FTIR Spectrometer. The analysis spanned the range of 4,000–600 cm^{-1}.

Thermal degradation characteristics of the cured samples were investigated *via* thermogravimetric analysis (TGA), utilizing a Perkin Elmer (TGA 4000). TGA measurements were conducted in a nitrogen environment, spanning temperatures from 30°C to 900°C, with increments of 10°C per minute.

Electrical volumetric and surface resistivity measurements were performed using an electrometer (ASTM D257). The average thickness of each sample was taken into account for volumetric resistivity measurements. During the volumetric resistivity measurement, the conditions were as follows: temperature of 25°C, applied voltage of 500 V, and measurement duration of 10 s.

3 Results and discussion

An overview of the key properties of the utilized monomers, encompassing molecular weight, density, flash point, and molecular structures is given in Table 2. Notably, VT exhibits a cyclic structure with a molecular weight of 118 $g{\cdot}mol^{-1}$, while BDDMA presents a long-chain structure with a higher molecular weight of 226 $g{\cdot}mol^{-1}$. Furthermore, it is worth highlighting that BDDMA possesses a significantly elevated flash point. The influence of these distinctive characteristics was thoroughly investigated through a series of characterization analyses.

Upon examining the results of the physical analyses presented in Table 3, it is apparent that the acid number and density values for UPE varnishes utilizing VT and BDDMA are closely aligned. However, in the realm of viscosity measurements, samples including BDDMA exhibited values approximately six times higher than those samples with VT (21). When comparing the viscosities of UPE-SA-VT and UPE-FA-VT, it is evident that the linear structure of the SA monomer (Table 2) has a substantial impact, resulting in viscosities almost twice as high as those of the FA-included samples. This disparity can be attributed to differences in molecular structure. The notably high flash point of 139°C associated with the BDDMA reactive monomer had a significant effect on the UPE-BDDMA varnish, resulting in a higher flash point when compared to the UPE-VT varnish (22). In consideration of the total solid content detailed in Table 1, it becomes evident that this aligns closely with the solid content observed when VT is employed in UPE synthesis. Conversely, there was an unexpected increase in solid content in UPE samples incorporating BDDMA. This distinction can be attributed to the fact that BDDMA is di-functional, whereas VT is mono-functional. This discrepancy is further substantiated by the differences in curing times, as explained in Section 2.3. Moreover, the %VOC

Table 2: Properties of monomers

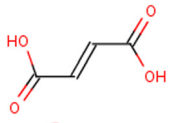
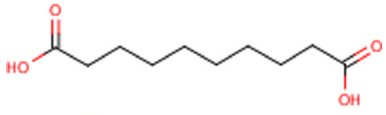
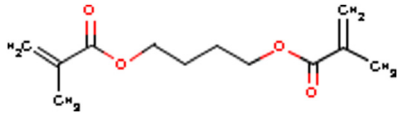
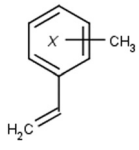
Monomer	Molecular weight ($g{\cdot}mol^{-1}$) (22)	Density ($g{\cdot}cm^{-3}$) (22)	Flash point (°C) (22)	Molecular structure (23)
FA	116.07	1.64	230	
SA	202.25	1.21	220	
BDDMA	226.30	1.02	139	
VT	118.18	0.89	52	

Table 3: Results of physical properties

Analysis	UPE-FA-VT	UPE-FA-BDDMA	UPE-SA-VT	UPE-SA-BDDMA
Acid number (mg KOH g^{-1})	14.40	17.00	16.00	17.50
Density (g·cm^{-3})	1.0435	1.1030	1.0328	1.0623
Viscosity (cP)	290.0	1770.0	520.0	1860.0
Solids content (%)	67.05	92.35	76.11	92.30
VOC (%)	4.30	2.30	3.60	2.30
Flash point (°C)	47.20	64.30	47.10	64.50

Table 4: Thermal analysis results of samples

Analysis	T_{d5} (°C)	$T_{dmax,\%50}$ (°C)	Residue amount (600°C, wt%)
UPE-FA-VT	234	388	10.0
UPE-FA-BDDMA	264	414	11.0
UPE-SA-VT	276	430	5.0
UPE-SA-BDDMA	270	436	4.0

content in the UPE-BDDMA samples consistently measured at 2.30%, falling within the specified range of 1–3% as per the literature (18). Importantly, this value was lower than that observed in the UPE-VT samples.

FTIR analysis was utilized to analyze the structural characteristics of the samples. In the FTIR spectrums of UPE-FA-BDDMA, UPE-FA-VT, UPE-SA-BDDMA, and UPE-SA-VT samples, all had C═O stretching vibration of ester group at 1,714, 1,714, 1,716, and 1,730 cm^{-1}, respectively. UPE-FA-BDDMA and UPE-FA-VT spectrums both had C-H stretching vibration at 771 cm^{-1}. UPE-FA-VT and UPE-SA-VT spectrums both had C═O stretching vibration of aromatic ester groups at 1,298 and 1,255 cm^{-1}. UPE-FA-BDDMA and UPE-SA-BDDMA spectrums both had C≈C stretching vibration at 1,635 cm^{-1} (23). FTIR spectra of all samples are shown in Figure 3.

In the existing literature, research on the thermal degradation of UPEs, resulting from the reaction of straight chain and branched chain glycols with MA and phthalic anhydride, isophthalic acid, or terephthalic acid, has been conducted using TGA (24,25). These studies have explored the impact of additional fillers, composition, and curing conditions on the thermal degradation of UPE, revealing that resins typically initiate degradation around 200°C and experience significant weight loss around 400°C. Notably, fillers and curing methods were found to have a limited effect on the stability of the resins. Furthermore, previous research (26) has established that the structure of the initiator can influence the thermal properties of UPEs (25,26). By using

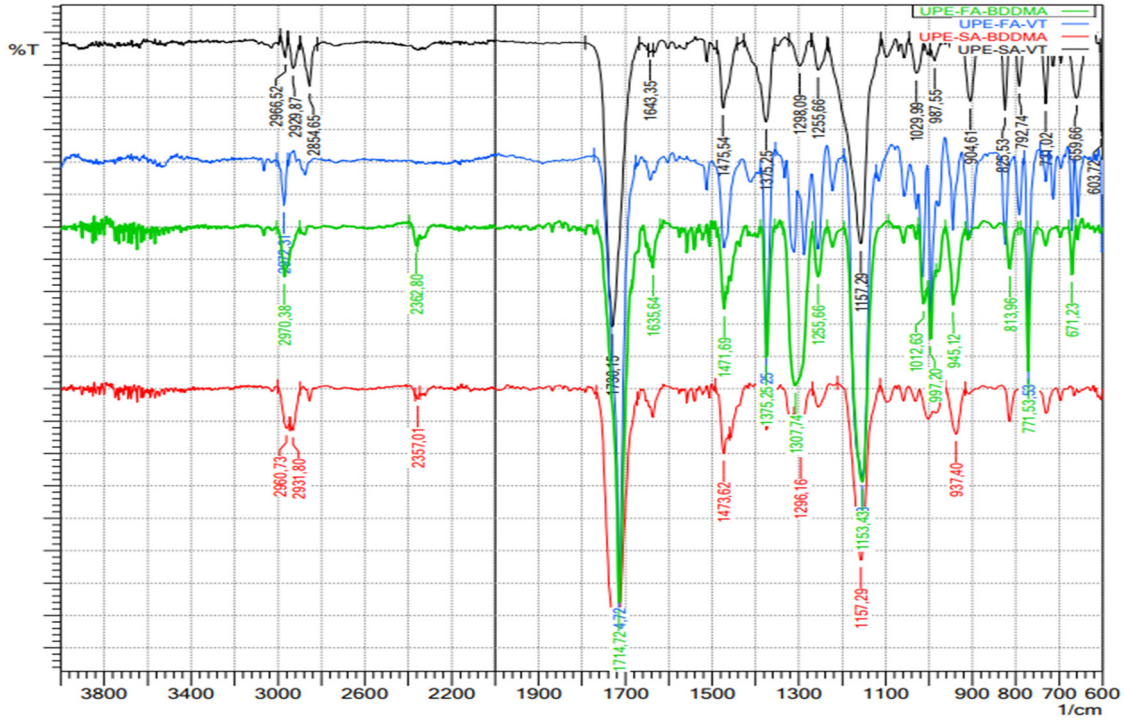


Figure 3: FTIR spectrums of samples.

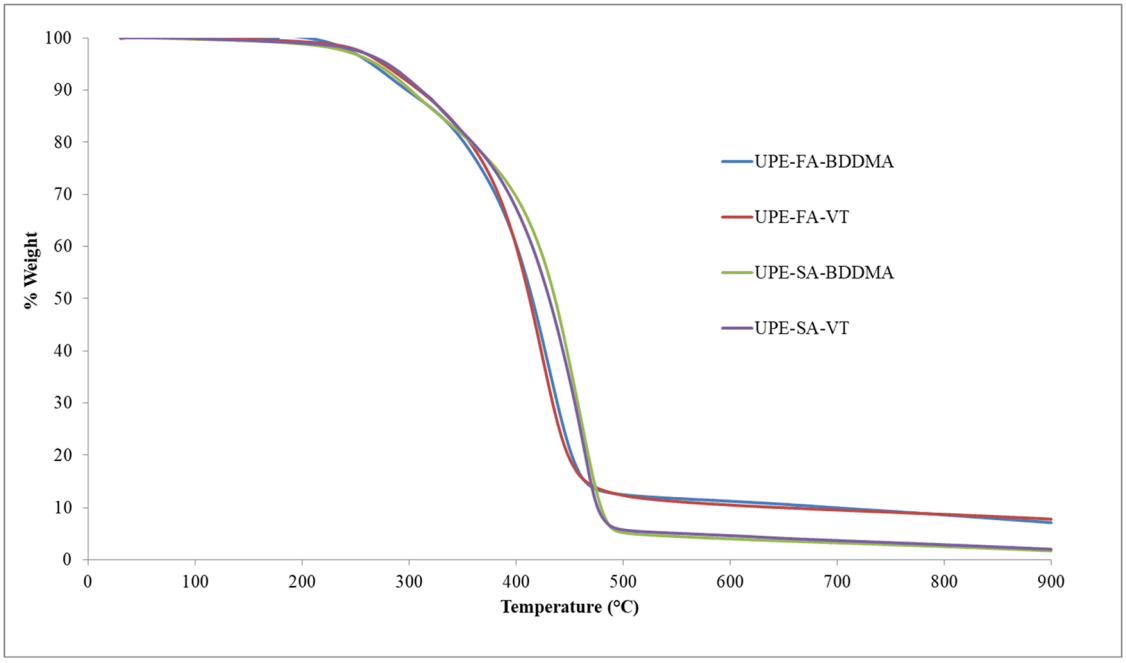


Figure 4: TGA graph of samples.

TGA, the decomposition initial temperature (T_{d5}), the maximum decomposition temperature ($T_{dmax,\ 50\%}$), and residue amounts at 600°C were measured and the results of TGA are summarized in Table 4 in this study. UPEs with decomposition temperatures in the range of 234–276°C were synthesized in accordance with the literature. When the reactive monomer was changed in UPE resin samples containing SA, there was not much change in the initial and maximum decomposition temperature, only a 6°C change was observed. In samples containing FA, the change in reactive monomer significantly affected the decomposition initial temperature. The UPE-FA-VT sample began to decompose at 234°C, while the UPE-SA-VT sample began to decompose at 276°C. The UPE-SA-BDDMA sample showed a high value with a maximum decomposition temperature of about 436°C. UPE resin containing SA show main degradation at higher temperatures than UPE containing FA. It was found that the most effective parameters affecting the decomposition temperature are monomer type and chemical structure. UPE-FA-BDDMA varnish showed the highest char residue of 11% at 600°C. In addition, as seen in the TGA graph in Figure 4, all the varnishes decomposed in a single step.

Analyzing the results presented in Table 5, it is evident that there are significant variations in electrical properties, particularly in electrical volumetric and surface resistivity measurements: For UPE-FA-VT, the electrical volumetric resistivity was measured to be 1.58×10^{15} Ω·cm, while for UPE-SA-VT it was 6.96×10^{11} Ω·cm. In terms of surface resistivity, UPE-FA-VT displayed a value of 1.96×10^{13} Ω, whereas UPE-SA-VT exhibited 7.47×10^{10} Ω. Unfortunately, it was not possible to obtain measurements for UPE-SA-BDDMA, as the varnish did not provide a suitable measurement surface after curing. Based on these findings, it can be concluded that varnishes incorporating the FA monomer tend to exhibit higher electrical resistance compared to those containing the SA monomer. However, it is notable that the choice of the reactive monomer does not seem to have a significant impact on these electrical properties. This disparity can be attributed to the presence of an unsaturated bond in the FA monomer's structure, which facilitates cross-linking. In contrast, the linear structure of the SA monomer does not allow for such cross-linking. In the case of the UPE-SA-VT sample, cross-linking is facilitated by other

Table 5: Electrical properties of samples

Analysis	UPE-FA-VT	UPE-FA-BDDMA	UPE-SA-VT	UPE-SA-BDDMA
Electrical volumetric resistivity (Ω·cm)	1.58×10^{15}	1.45×10^{15}	6.96×10^{11}	—
Electrical surface resistance 500 V(Ω)	1.96×10^{13}	2.79×10^{13}	7.47×10^{10}	—

compounds, such as MA and THPA, present in the structure. As a result, the use of the FA monomer provides an advantage over the SA monomer in terms of dielectric properties within UPE coatings.

4 Conclusion

The study involved the synthesis of UPE resins and a comprehensive evaluation of their physical, thermal, electrical, and structural characteristics through various analytical methods. Additionally, the investigation delved into the impact of different reactive monomers, specifically acid monomers, on the properties of the synthesized varnishes. The incorporation of the BDDMA reactive monomer resulted in notable improvements in viscosity and flash point of the varnishes when compared to VT. Furthermore, the UPE varnishes containing the SA acid monomer exhibited higher initial degradation and maximum degradation values in comparison to the FA sample. In the realm of electrical analysis, varnishes containing the FA acid monomer showcased superior dielectric (insulating) properties when contrasted with varnishes containing the SA acid monomer. This research underscores the significant influence that specific reactive monomers can have on the properties of UPE resins and the resultant varnishes, thereby enhancing various aspects such as thermal stability, viscosity, and electrical insulating properties. As a result, our approach holds the potential to pave the way for the creation of innovative UPE resins with customizable properties, suitable for a wide spectrum of applications across diverse fields.

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Author contributions: (https://casrai.org/credit/). Selinay Gümüş: conceptualization, methodology, resources, formal analysis, data curation, writing – original draft, and visualization. Kaan Aksoy: conceptualization, writing – review and editing, and supervision. Ayşe Aytaç: conceptualization, methodology, formal analysis, data curation, writing – original draft, writing– review and editing, and visualization.

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