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Effect of Functional Carbon Nanotube Fillers on the Physical and Thermal Characterization of Radiation Curable Coating

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Abstract: This work investigates the formulation of a radiation-curable coating that integrates functionalized carbon nanotubes (f-CNT) to improve surface characteristics. Trimethylolpropane triacrylate (TMPTA) was employed as the monomer, with epoxy acrylate and urethane acrylate as oligomers and Irgacure-500 as the photoinitiator. To cure the coatings, the formulations were subjected to ultraviolet (UV) light, and the surface properties were investigated using Fourier-transform infrared spectroscopy (FTIR), pendulum hardness testing, and thermogravimetric analysis (TGA). The results show that altering the ratios of f-CNT in the formulations have a considerable impact on the mechanical and thermal characteristics of the coating. The findings show that ideal compositions of these additives improve the hardness, crosslinking density, and thermal stability of the cured films, offering insight into their potential.

Keywords: Functionalized carbon nanotubes (f-CNT); Radiation-curable coatings; UV curing

Introduction

Radiation curing is a process of coating, ink, or adhesive polymerizing (crosslinking) directly on a substrate through interaction with incident radiation. Crosslinked or cured is facilitate by exposing the coating with high-intensity radiation energy from electron beams or ultraviolet light radiation that will harden the coatings. Unlike traditional curing processes that rely on thermal activation, radiation curing enables faster production rates and provides distinct advantages in terms of energy consumption, product performance, and environmental impact. Because of the near-instant cure and lack of solvents, a dry, fully cured film may be obtained in a matter of seconds (Harun et al., 2018). One of the reasons for the rising relevance of UV-curing methods in both industry and academic research is a unique property that causes the rapid transformation of a liquid monomer into a solid polymer film with different physical-chemical and mechanical characteristics (Sangermano et al., 2018).

UV-curable coating also is becoming a popular alternative to solvent-based coating due to its environmental benefits. This procedure is environmentally friendly as it uses no solvents and is often performed at ambient temperature, resulting in energy savings. This method also does not require the use of volatile organic solvents which leads to significant reductions in volatile organic compound (VOC) emissions, aligning with modern industry's push toward greener, more sustainable manufacturing practices (Patil et al., 2023). Furthermore, efficient, environmentally friendly, energy-saving, enabling, and inexpensive technologies are regarded as the 5E benefits of UV curing technology. UV-curing generates radical or cationic species by contact with a photoinitiator. This

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leads to radical chain-growth or cationic ring-opening polymerization. When exposed to UV light, most monomers do not create significant quantities of initiating species, necessitating the addition of a photoinitiator to the formulation. Once initiated, the chain reaction proceeds at a faster pace than in traditional heat polymerization. In general, UV-irradiated epoxy resins cure by cationic polymerization.

The main components of radiation curing are monomer, oligomer, photoinitiator and additives. The monomer used in this project is Trimethylolpropane Triacrylate (TMPTA) offers high reactivity and fast curing speed due to its high double bond content and excellent crosslinking ability, improving hardness, gloss, chemical and wear resistance of the final product. The addition of the additive which is functionalized CNT (f-CNT) intended to see its effect on the surface of the coatings. It is commonly known that CNTs and its derivative, graphene typical carbon nanomaterials having 1D fibrous and 2D plate structures, respectively. Both nanomaterials have been extensively studied for their potential to enhance mechanical strength, conductivity, and barrier properties when integrated into polymer matrices. The objective of this study is to investigate the characteristics of UV-curable TMPTA-based coatings modified with f-CNTs. The findings will contribute valuable insights into the design and development of advanced coatings for a wide range of industrial applications.

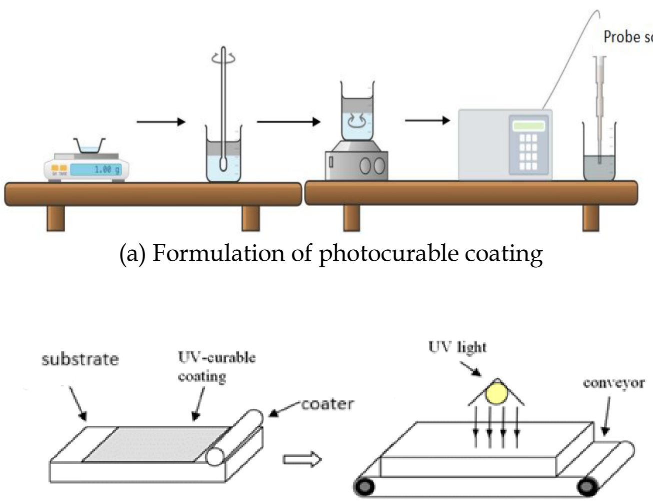
Method

Materials

The preparation of the formulation involves four main components of material which are TMPTA as a monomer, Epoxy acrylate (EB600) and Urethane acrylate (EB210) as oligomers, perfluorodecyl acrylate (PFOA) as hydrophobic agent and Irgacure 500 (IC-500) as the photoinitiator. All formulations have a fixed weight of TMPTA, EB600, EB210 and IC-500. The various compositions of f-CNT was incorporated to study the effects on the final cured coatings. The composition of the materials is shown in Table 1 below whereas the preparation setup can be found in Figure 1.

Table 1. Composition of materials in the radiation curable formulation with different loadings of f-CNT

Sample	PFOA (%)	IC 500 (%)	EB 210 resin (g)	EB600 resin (g)	TMPTA (g)	f-CNT (%)
TMPTA- PFOA	0.6	5	1	2	10	0
0.05 % f-CNT	0.6	5	1	2	10	0.05
0.1 % f-CNT	0.6	5	1	2	10	0.10
0.2 % f-CNT	0.6	5	1	2	10	0.20
0.3 % f-CNT	0.6	5	1	2	10	0.30



(a) Formulation of photocurable coating

(b) UV irradiation of the curable coating

Figure 1. A setup for UV curable coating film preparation and irradiation

Characterization

The IR spectra for the cured coating films were observed using FTIR spectrophotometry (Bruker Tensor II, Spectrum 2000). All spectra were obtained using the attenuated total reflection (ATR) method. A total of 32 scans were collected within a wavenumber range of 500–4000 cm−1, with a resolution of 4.0 cm−1 for all recorded spectra. The hardness of the UV curable coating film was conducted by using a pendulum hardness tester TQC Sheen from Netherlands and follows ISO 1522 standard method, namely König test method. The coating's hardness performance was calculated using the percentage of pendulum hardness oscillation (PH%) in relation to the glass standard. The wetting characteristic was measured using an optical contact angle (CA) device from Biolin Scientific (Finland). A measurements for distilled water droplets were recorded for each prepared sample. For CA inspection, images of 5 µL droplets of distilled water that were horizontally placed on the sample holder were captured to determine the static CA. The contact angle values for water droplets on the left and right sides of the curable coating surface, with an accuracy of 0.1°, were determined using the Young-Laplace fitting method with Attension Theta software. All measurements were performed in triplicate at an room temperature of 30°C. The thermal analysis was measured using TGA model TG 209 F3 Tarsus (Netzch). The thermogravimetric analysis (TGA) records the weight fluctuation, which is the weight loss during heating as a function of temperature corresponding to the evolution of volatile compounds, thermal degradation of the material.

Result and Discussion

FTIR result

The functional groups of TMPTA, and f-CNT were determined by FTIR spectroscopy and results are shown in Figure 2. In general, all infrared spectra did not have a distinct difference between each coating formulation. This is due to the major presence of TMPTA in all the formulation. At 2965 cm^{-1} the peaks belong to the C-H stretching vibrations which shows the presence of alkyl group in TMPTA (Ismail et al., 2021). The peak characteristics at 1718 cm^{-1} assigned to the C=O stretching vibrations describes the presence of ester group. At a wavenumber between 1617 cm-1 to 1636 cm^{-1} describes the characteristics of C=C stretching vibrations indicating the presence of alkenyl group in TMPTA. Then, at 1405 cm^{-1} there is significant peaks belonging to C-H bending vibrations that confirming the alkyl group. Between 1060 cm-1 to 1200 cm^{-1} the peaks characteristic for C-O stretching vibrations indicating the presence of ether group. At 3450 cm^{-1}, there is a broad peak of O-H stretching describe the presence of hydroxyl (-OH) groups on the surface of f-CNT. Peak at 1630 cm-1 corresponds to the C=O stretching vibration, indicating the presence of carboxyl (-COOH) groups and peak at 1720 cm^{-1} and 1040 cm^{-1} it corresponds to C=O and C-O stretching vibrations, respectively confirming the presence of carboxyl group in f-CNT (Salihu Adamu Girei et al., 2011). The peak characteristic of f-CNT not really have a major difference and the difference should be significant to infrared spectra of cured sample (Sardinha et al., 2019).

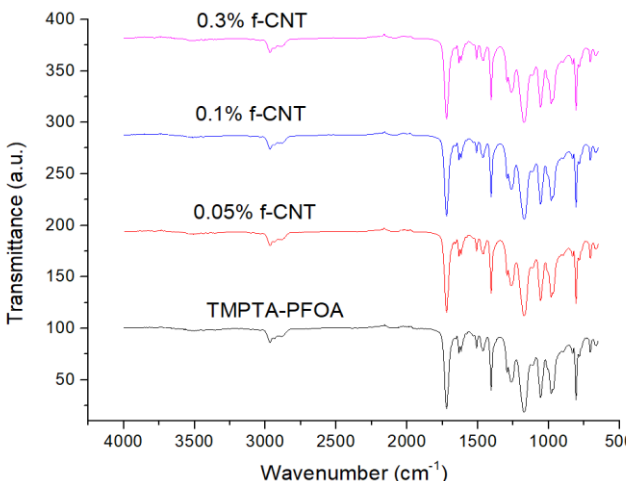


Figure 2. FTIR spectra for TMPTA-PFOA resin containing different loading of f-CNT

Water Contact Angle (WCA) Test

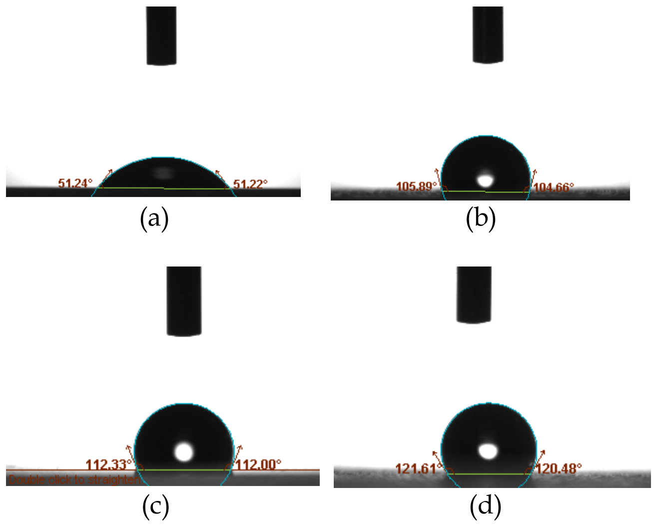


Figure 3. Contact angle reading of (a) bare glass, (b) TMPTA-PFOA, (c) 0.05% f-CNT and (d) 0.3% f-CNT

The water contact angle represents the wetting property of TMPTA formulation can be found in Figure 3. Figure 3(a) shows the WCA of a typical bare glass which is hydrophilic and the contact angle is 51.2°. A significant increase of the contact angle obtained (Figure 3(b) for TMPTA-PFOA at 105.8° due to hydrophobic property of the film. This property is crucial in real application where it improves surface protection due to dirt, corrosion and rain. The hydrophobic property is attributed to PFOA characteristic that is water proof. Figure 3 (c) and (d) shows f-CNT loading TMPTA coating film. The addition of f-CNT increases the surface roughness of the coating film and in subsequent increase the WCA reading where the reading taken for both films is 112.3° for 0.05% f-CNT and 121.6° for 0.30% f-CNT respectively (Harun et al., 2018).

Pendulum Hardness Test

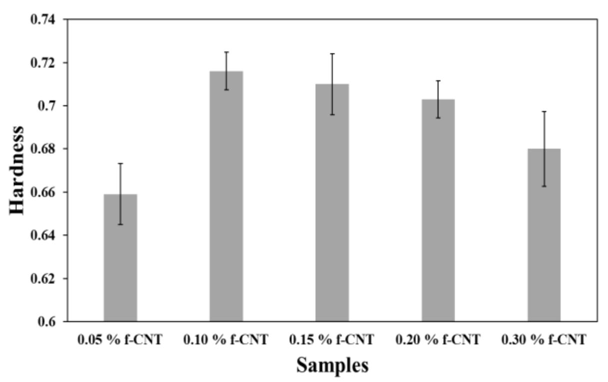


Figure 4. Pendulum hardness of UV curable coating film at different f-CNT loadings

The hardness and elasticity of coatings after irradiation was studied by Pendulum Hardness tester using König test. The hardness of each coating is shown in Figure 4. Samples with different percentage of f-CNT affected the hardness of each sample. Samples with 0.10 % of f-CNT has the highest hardness (0.716) and samples with 0.05 % of f-CNT has the lowest hardness. The hardness trend increases significantly from 0.05% to 0.10% of f-CNT and gradually decrease from 0.10 % to 0.30 % of f-CNT. Samples with 0.15 % of GO has the highest hardness and sample with 0.3 % of GO has the lowest hardness. The sample with high hardness indicates that the sample have more toughest surface (Alias et al., 2023). This may describe it have more crosslinks in the cured coating (Said et al., 2013).

Thermogravimetric Analysis

The thermal stability and thermal degradation of cured coating was studied by TGA analysis with temperature range from 35 ℃ to 900 ℃. In Figure 5 below, it shows the TGA thermograms of the TMPTA film surface from cured coatings. All thermograms exhibits two stages of thermal degradation. The thermograms possess a same characteristic of thermal degradation from 35 ℃ to 400 ℃. The mass loss occurs in this region due to the loss of volatile compounds or photoinitiator in the films. Coating film shows a mass loss between 60 ℃ to 100 ℃ may be due to the evaporating of water contained in the coating's films, indicating that there is present of moisture at the films. A significant drop of the mass loss peaks was shown in every thermograms. This major degradation occurred between 400 ℃ to 500 ℃ due to the decomposition of organic polymer chains (Hatanaka et al., 2017). The temperature range between 500 ^{o}C - 900^{o}C distinguishes the steady weight loss due to the decomposition of carbon skeleton within TMPTA formulation (Alias et al., 2023).

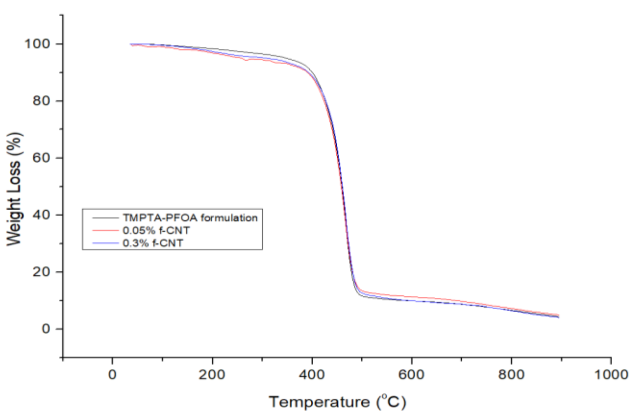


Figure 5. TGA curves of UV curable coating film of TMPTA-PFOA film and different f-CNT loadings

Conclusion

The work effectively illustrates that the integration of f-CNT into UV-curable TMPTA-based coatings may markedly improve the mechanical and thermal characteristics of the cured films. FTIR analysis showed the existence of functional groups associated with the additives, while the pendulum hardness test demonstrated that PFOA at a percentage of 0.6% greatly increases the hardness of the coating on glass. This shows that it has the most durable coating and the same as that of 0.10% f-CNT. The wetting properties increase when the f-CNT is added due to the roughness present in the coating films. TGA analysis analyzes the temperature behaviour of organic polymer chains, among other volatile compounds in the formulations. These findings indicate that this innovative formulation has significant promise for use in advanced coating applications, particularly those that need high mechanical strength and thermal stability.

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Author Contributions

Conceptualization, M. H. H, M. H and M. S. A; methodology, K. A. K.; software, N. O and K. A. K.; validation, M. H. H, M. H, R. T., M. F. A. R. and M. S. A; formal analysis, K. A. K, I. M. Z. and S. N. E. W. M. A.; investigation, K. A. K, I. M. Z. and S. N. E. W. M. A.; resources, M. H. H.; data curation, K. A. K, I. M. Z., K. N. K. U. and S. N. E. W. M. A.; writing—original draft preparation K. A. K. and M. H. H.; writing—review and editing, M.H. and M. S. A.; visualization, M. H. H. and M. S. A; supervision, M. H. H and M. S. A.; project administration, M. S. A. and M.H. H.; funding acquisition, M.H.H. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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