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Polyaniline and Its Role on Thermal Stability, Viscoelastic Behavior and Electrical Conductivity of UV Curable Epoxy Resin

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Abstract

Ultraviolet (UV) curing systems have garnered significant attention and scholarly focus due to their remarkable ability to facilitate rapid curing processes while simultaneously exhibiting low energy consumption characteristics, which are increasingly pertinent in contemporary manufacturing and environmental sustainability discussions. The aim of this study is investigating the multifaceted role that polyaniline in UV-curable epoxy acrylate coatings. Both polyaniline and epoxy acrylate resin were synthesized through established chemical processes and subsequently characterized using a range of analytical techniques. To assess the impact of polyaniline on the epoxy acrylate resin, varying percentages of polyaniline, specifically at concentrations of 1%, 3%, and 5%, were systematically incorporated into the epoxy acrylate resin matrix to evaluate their effects on the resultant material properties. A detailed analysis of the morphological

characteristics of the prepared nanocomposites revealed a well-distributed and appropriate dispersion of polyaniline particles throughout the polymer matrix, which is crucial for achieving optimal performance in the final product. Thermal stability of the sample containing 3% polyaniline exhibited a remarkable enhancement, being twice as high when compared to the sample that contained only 1% polyaniline, thus highlighting the significance of polyaniline concentration on thermal properties. Furthermore, it was observed that the electrical conductivity of the sample incorporating 3% polyaniline experienced a substantial increase, quantified at an impressive factor of 4.25 times greater than that of the sample with a lower concentration of 1%, underscoring the enhanced electrical performance attributable to the optimized incorporation of polyaniline within the epoxy acrylate matrix.

Keywords: Epoxy acrylate, Polyaniline, Thermal stability, Viscoelastic behavior

1. Introduction

One of the common resins in the paint and coatings industry is epoxy resin, which has many applications due to its unique properties [1-3]. Epoxy resin is a two-component resin and needs a suitable hardener for curing. The complete curing time is from a few hours to a few days, which can be accelerated by heating [4-7]. Although this process is accepted and used until now, in some cases the long curing time causes problems that the use of ultraviolet violet curing (UV-curing) systems is a suitable solution [8-11]. Increasing attention to UV curing systems due to the solvent-free system, quick curing, and low energy consumption has led to the creation of new applications for it and its use in various research and industrial sectors [12-15]. Many methods have been proposed in

the field of the synthesis of UV curable resins, and in general, the synthetic processes are by acrylated resin with the help of suitable raw materials, which have a carbon-carbon end double bond and a catalyst [16-20].

A group of polymers such as polyacetylene (PA), polyaniline (PANi), polypyrrole (PPy) and polythiophenes (PTs) have a special structure that conducts electric current to pass through them and are known as conductive polymers. Blending conductive polymers with other polymers can lead to mixtures with special properties for specific applications. In fact, conductive polymers in the matrix of other polymers act like ways for the passage of electric current, the more these ways there are, the greater the electrical transmission ability. The electrical conductivity of conductive polymers can be changed by doping them or adding nanoparticles [21-24]. Polyaniline, celebrated for its unique features, is one of the most popular conductive polymers. It has a suitable performance for various applications including surface coatings. This polymer offers advantages such as excellent adhesion properties and good chemical stability and is suitable for coating on various surfaces [25, 26]. If polyaniline is used in nano dimensions, it can have different functions in the polymer substrate. Therefore, the distribution of polyaniline nanoparticles and their dimensions are effective in the electrical conductivity of the nanocomposite [27-29]. Polyaniline has the ability to perform reversible redox reactions and can act as a semiconductor and a conductor. For this reason, it is used as an anti-corrosion additive in coatings and for anti-static coatings and electromagnetic shielding applications [30, 31].

Based on a comprehensive review of existing literature, the incorporation of polyaniline within the matrix of epoxy acrylate resin remains unexamined, thereby necessitating

further investigation in this specialized field. The primary objective of this research was to investigate strategies for the formulation of UV-curable epoxy acrylate nanocomposite coatings that incorporate polyaniline nanoparticles, with the intent of producing coatings characterized by rapid curing and economic efficiency. Furthermore, an evaluation of particle distribution has been performed through the examination of morphological characteristics, electrical conductivity, and viscoelastic properties. The findings of this research may be advantageous in the development of sensors, UV-curable conductive adhesives, and coatings with corrosion resistance.

2. Experimental

2.1. Materials

Acrylic acid, triphenylphosphine, hydroquinone, aniline, ammonium persulfate, hydrochloric acid were obtained from Merck Co. (Germany). Epoxy resin KER 828 with a viscosity13 Pa.s, at 25°C and EEW 185-190 g/eq. was purchased from Kumho Co. (Korea). Moreover, diphenyl (2,4,6-trimethylbenzoyl)- phosphine Oxide (TPO), acting as a photoinitiator was sourced from Sigma-Aldrich Co. (USA).

2.2. Synthesis of epoxy acrylate resin

One mole of epoxy resin was added to a 500 ml three-necked round-bottom flask equipped with a mechanical stirrer, nitrogen inlet and drop funnel. The temperature of the balloon was elevated to 70°C using an oil bath. An amount of 0.1% triphenylphosphine as a catalyst and 0.1% hydroquinone as an inhibitor was added to the flask and stirring was continued until a homogeneous mixture was reached. After homogenization, 2 moles

of acrylic acid were added to the mixture by a drop funnel for 30 minutes, then the temperature rose to 90°C and the reaction was persisted for 4 hours.

2.3. Synthesis of nano polyaniline

Polyaniline was prepared by oxidative polymerization of aniline in the presence of ammonium persulfate. In the first step, 2 ml of distilled aniline was stirred in 100 ml of 1 M hydrochloric acid solution for half an hour at room temperature. Then 40 ml of 1 M ammonium persulfate solution was gradually introduced to the reaction container and mixed for 5 hours at 0 °C temperature, then the reaction mixture was kept in the refrigerator for one day to complete the reaction. After filtering the sediment obtained, it was repeatedly washed with an aqueous solution of hydrochloric acid and dried in an oven for 10 hours. The obtained material was the conductive form of polyaniline (PANi) in emeraldine form.

2.4. Formulation of nanocomposites

Ratios of 1, 3 and 5 wt.% of synthesized polyaniline were added to epoxy acrylate resin. In the first step, the samples were mixed using sonication three times for 10 minutes to separate the polyaniline agglomerates and disperse them well in the epoxy acrylate resin. Then, 3 wt.% of Diphenyl (2,4,6-trimethylbenzoyl)-phosphine oxide (TPO), as a photonitiator, was added to each sample and mixed with a magnetic stirrer until complete mixing. Polymer films with a thickness of 120 microns were applied on a steel substrate with a film applicator and cured using a UV-LED lamp for 1 minute.

Table 1. Naming samples and their percentage composition.

Sample	Epoxy acrylate (%)	TPO (%)	Polyaniline (%)
Blank	97	3	0
1% PANi	96	3	1
3% PANi	94	3	3
5% PANi	92	3	5

2.5. Characterization

Fourier transform infrared (FT-IR) spectra were recorded on the Perkin-Elmer spectrophotometer, Spectrum 1 model (USA). The morphology was studied by PHILIPS scanning electron microscope (SEM) XL30 FEG. The viscoelastic behavior was studied by dynamic mechanical thermal analyzer (DMTA), Netzsch, DMA 242 C (Germany). To conduct electrical conductivity, four-point conductivity test was performed according to the C611ASTM standard using the American-Precision Company's multimeter, model A5491, and the Meter Squrgedc (ITECH) company's power supply from China.

3. Results and Discussion

3.1. FTIR spectroscopy

Figure 1 shows the FTIR spectrum of polyaniline. The peak area of 3443 cm^{-1} is characteristic of N-H in the structure and the peaks of 2963 and 2927 cm^{-1} are related to aromatic C-H. The peaks in 1638 and 1472 cm^{-1} correspond to the C=C bond in the quinone and benzoic rings, respectively. Also, the peaks at 1294 and 1107 cm^{-1} indicate C-N in benzenoid. The peak area of 804 cm^{-1} also characterizes the C–H bond.
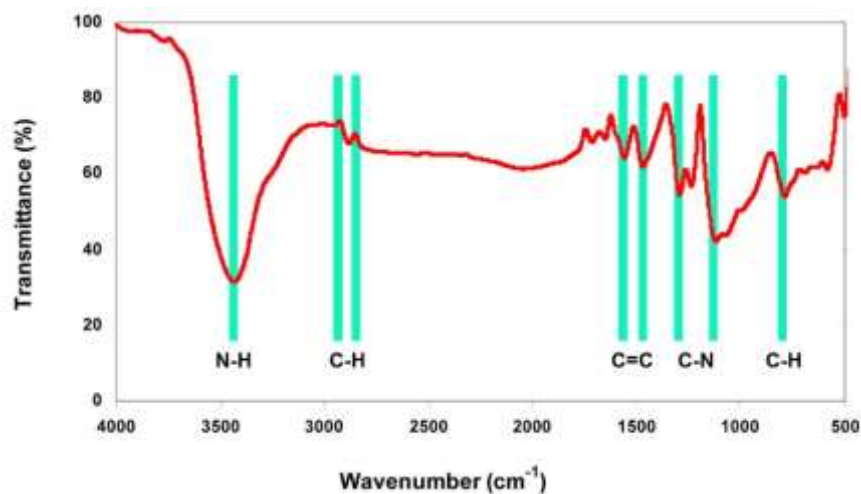
Figure 1. FTIR spectrum of polyaniline

Figure 2 shows the spectrum of the synthesized epoxy acrylate. Within this Figure, the peak observed at 3475 cm^{-1} corresponds to the elongation vibration of the hydroxyl group (OH), whereas the peaks at 3104 and 2962 cm^{-1} are associated with the stretching vibration of aromatic and aliphatic C-H bonds, respectively. A carbonyl group (C=O) is evident at 1723 cm^{-1}, and a C=C bond is present at 1510 cm^{-1}, along with the C-O bond of the acrylate unit ether at 1043 cm^{-1}. Consequently, it can be deduced that the epoxy acrylate resin has been successfully synthesized.
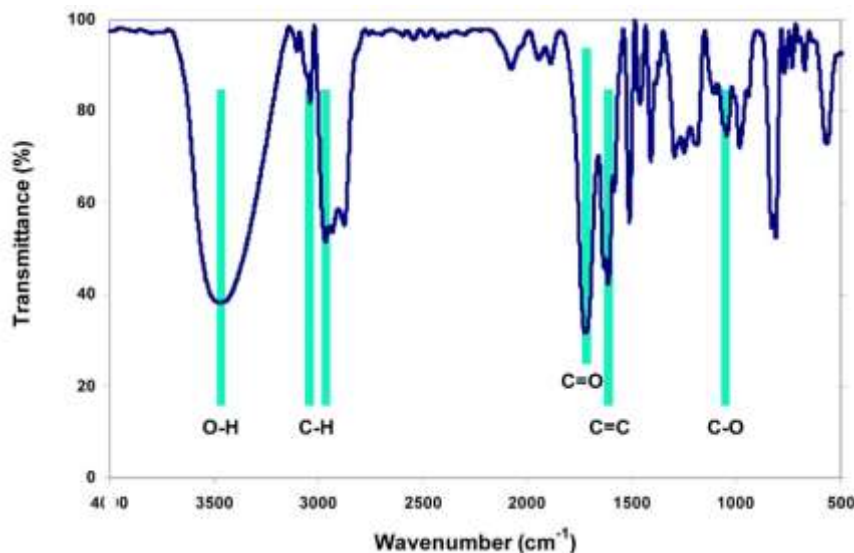
Figure 2. FTIR spectrum of epoxy acrylate resin.

3.2. Morphology

Figure 3 shows the FE-SEM images of dispersed polyaniline (PANi) in an epoxy acrylate matrix. Samples, a, b and c contain 1, 3 and 5% polyaniline, respectively. The results of the microscopic images indicate that the polyaniline particles are well dispersed in the epoxy acrylate matrix. In the sample containing 1% polyaniline, the particles are distributed homogeneously and without aggregation in the polymer matrix. With increasing the amount of polyaniline, a slight increase in the form of local aggregations is observed in the sample containing 3 % polyaniline. In the sample containing 5% polyaniline, an increase in the aggregation of polyaniline particles is observed. The formed aggregations can be attributed to the intrinsic properties of polyaniline and epoxy acrylate in terms of polarity. The conjugated structure of polyaniline has a rigid backbone. When introduced into a flexible matrix such as epoxy acrylate, the rigid chains tend to be placed together instead of uniformly dispersed and aggregations are formed. On the other hand, as the amount of polyaniline increases, the viscosity increases and

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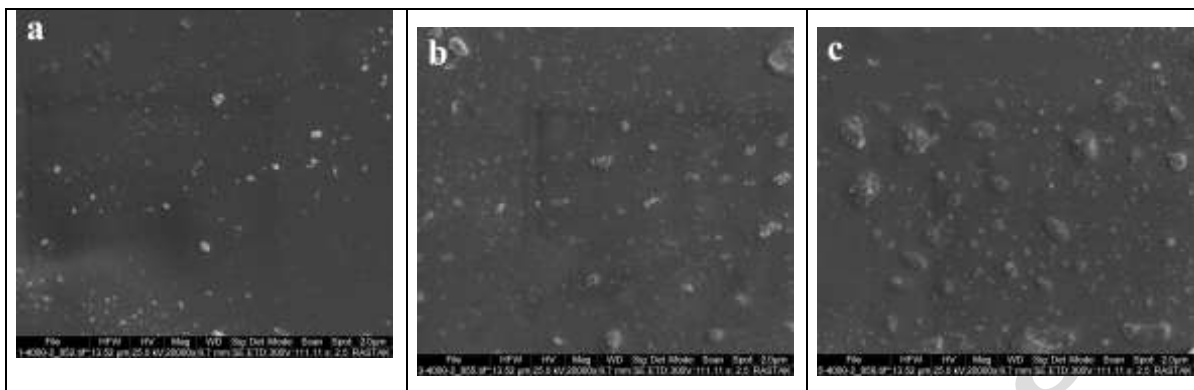mixing becomes more difficult and particle aggregations occur [32].

Figure 3. SEM images of epoxy acrylate/polyaniline nanocomposites. a: 1%, b:3% and

c:5%.

3.3. Electrical conductivity

Figure 4 shows the electrical transmission ability of the prepared nanocomposite

coatings. As it is known, with the increase in the amount of PANi, the electrical

transmission has increased. The quantity of this parameter in samples 1, 3 and 5% PANi

is 2.09×10^{-5}, 3.85×10^{-5} and 1.64×10^{-4} S/cm, respectively.

Polyaniline is an intrinsically conductive polymer that can form continuous conductive

networks in an epoxy matrix. When added at low percentages, it may be close to the

diffusion threshold, forming the primary electron transport pathways. However, these

pathways are sparse, leading to a modest increase in conductivity. As the polyaniline

concentration increases, particle contact increases, forming more efficient continuous

conductive pathways. At 5%, the electrical conductivity increases as the interconnecting

networks become more complete. On the other hand, polyaniline doped at low

percentages carries a limited number of charge carriers, leading to a modest increase in

conductivity. Further additions of doped polyaniline (5%) have a higher electron density and maximize conductivity compared to 1 and 3% polyaniline. The conductivity of the sample containing 5% polyaniline is 4.25 times that of the 3% sample. The reason for this sudden increase is that it follows a diffusion model of electrical conductivity in composite materials. which increases dramatically once the critical concentration of polyaniline is reached. For this reason, for a sample containing 1% polyaniline, there are only a few conductive paths, which leads to a small increase in conductivity. In the 3% polyaniline sample, it is probably above the diffusion threshold, causing an increase in conductivity as the polyaniline particles are more closely connected and form a network. However, in the 5% polyaniline sample, due to the increased network, it is above the diffusion threshold, and the conductivity reaches its maximum achievable level [33, 34].
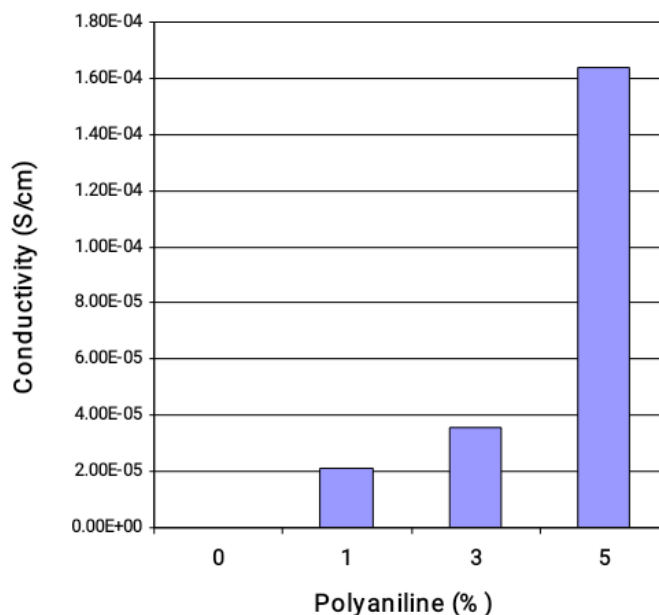

Figure 4. Comparison of electrical conductivity of epoxy acrylate coating with different percentages of PANi.

3.4. Thermal stability

For the purpose of studying the thermal stability of prepared nanocomposites, a thermogravimetric test was used, and the DTG diagram is shown in Figure 5. As shown in figure 5, the losing weight for all samples takes place in three steps. The first stage takes place in the temperature range of 120-200 °C, which is related to the moisture present in the samples and unreacted materials. The second step of weight diminution is seen at the temperature range of 260-380°C, which is related to the degradation of polyaniline. With increasing temperature, the third stage of degradation takes place from 400-500 °C, in which epoxy acrylate resin is destroyed. It is also clear that the thermal stability increased with the increase of PANi amount, which can be explained by the intermolecular forces of PANi and epoxy acrylate [35-37].

A fall in the area under the graph in the DTG diagram indicates the formation of more stable compounds and a decrease in the liberation of volatile substances and the speed of the degradation reaction. As illustrated in Figure 5, the area under the curve decreases with escalating amounts of polyaniline and this indicates the stability of the nanocomposite, which increases due to the increase in polyaniline and its interactions with the epoxy acrylate polymer chains the degradation more difficult and increasing the thermal stability [38].

The addition of polyaniline to epoxy acrylate resin has increased thermal stability due to the creation of bonds between nanoparticles and polymer chains. The more these bonds, the higher the thermal stability, and therefore, with increasing polyaniline content, thermal stability has increased and the peak area under the curve has decreased, indicating increased thermal stability. Another factor that can be attributed to the increase

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in thermal stability and the decrease in the area under the curve is the strengthening of the polymer network and the reduction in the mobility of the polymer chains due to the presence of more polyaniline [39, 40].
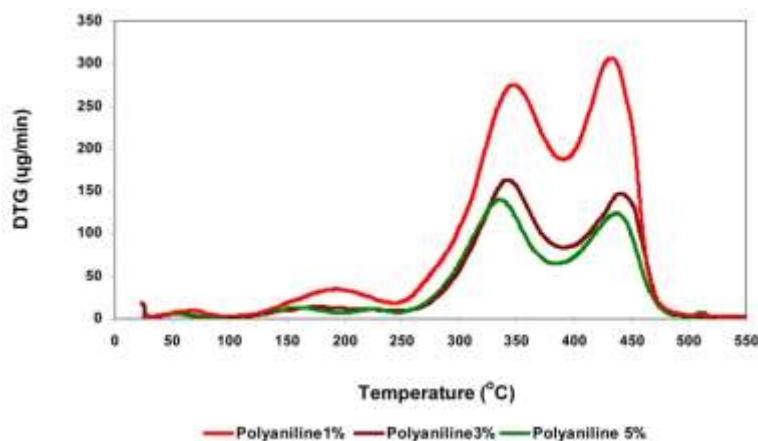

Figure 5. DTG diagram of epoxy acrylate/polyaniline nanocomposites.

3.5. Viscoelastic behavior

Figure 6 presents the elastic modulus (E') of resin samples composed of epoxy acrylate and polyaniline with varying proportions. The elastic modulus, also known as the modulus of elasticity or Young's modulus, is a measure of a material's stiffness and its ability to deform under stress. At lower concentrations of polyaniline (1%), the elastic modulus of the cured nanocomposite may experience a moderate increase compared to the pure epoxy acrylate. Polyaniline, as a conductive polymer, can elevate the mechanical performance of the composite by providing additional reinforcement [41]. It forms a network within the epoxy matrix, improving stiffness and strength. With a higher concentration of polyaniline (3%), there may be a more significant decrease in the elastic modulus, attributable to the correct distribution of polyaniline particles in the epoxy matrix.

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The addition of polyaniline at this level enhances the interfacial interactions between the polymer matrix and the nanofiller. This improved compatibility can lead to better load transfer between the polymer matrix and the PANi, resulting in increased stiffness. At even higher concentrations of PANi (5%), there might be a plateau or a slight decrease in the elastic modulus.

Beyond a certain concentration, the excessive amount of PANi might lead to agglomeration or clustering of the nanofillers, causing uneven distribution within the matrix. This clustering could create stress concentrations and defects, leading to a reduction in the overall stiffness.

Polyaniline, being a conductive polymer, can act as a reinforcing agent, enhancing the mechanical properties of the composite [42]. The concentration of PANi affects the quality of interfacial interactions between the polymer matrix and the nanofiller. Higher concentrations generally lead to better load transfer. At higher concentrations, the risk of agglomeration increases, causing uneven dispersion of PANi and potential negative effects on mechanical properties. There might be an optimal loading percentage where the positive effects on stiffness are maximized before diminishing returns or negative effects set in.
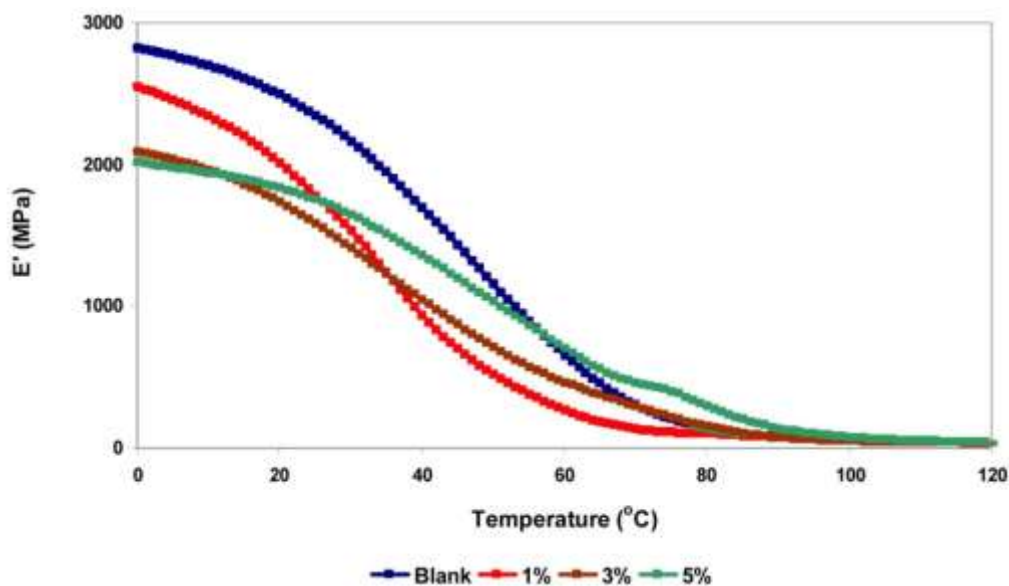
Figure 6. Elastic modulus of epoxy acrylate/polyaniline nanocomposites.

The loss modulus quantifies energy dissipation or damping characteristics of a material when subjected to deformation. As can be seen in Figure 7, at low temperatures, the loss modulus decreases with increasing polyaniline in the polymeric matrix because there are fewer nanoparticles and less molecular motion, and as a result, there is less mobility due to the friction between the polyaniline and the epoxy chain, so energy loss is reduced. Furthermore, at low temperatures, the thermal activation energy for molecular motion is usually higher, which limits motion and energy dissipation. At higher temperatures, the loss modulus increases due to the increased molecular mobility of polyaniline nanoparticles, which leads to increased friction and energy loss. In addition, at higher temperatures, the possibility of thermal excitations and molecular motion increases, which leads to more energy loss and thus an increase in the loss modulus [43]. In addition, at high temperatures, structural changes such as polymer chain rearrangement or nanoparticle dispersion may occur, leading to changes in mechanical properties and

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consequently to changes in the loss modulus [44].

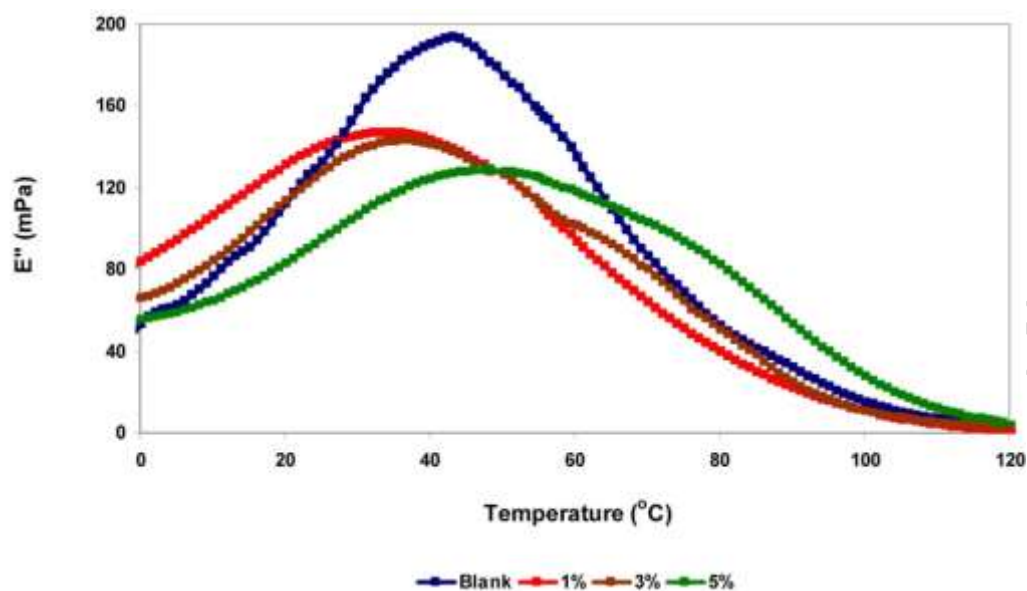**Figure 7.** Loss modulus of epoxy acrylate/polyaniline nanocomposites with different percentages of polyaniline.

Figure 8 shows the diagram of tanδ for epoxy acrylate/polyaniline nanocomposites with different percentages of polyaniline. The presence of PANi in epoxy resin increased the crosslink density by increasing the intermolecular interactions, and it is expected that the glass transition temperature also increases [45]. According to the results and as can be seen, the glass transition temperature shows an increase and for the blank sample, 1, 3 and 5% PANi are equal to 77, 80, 87, and 93 °C respectively.
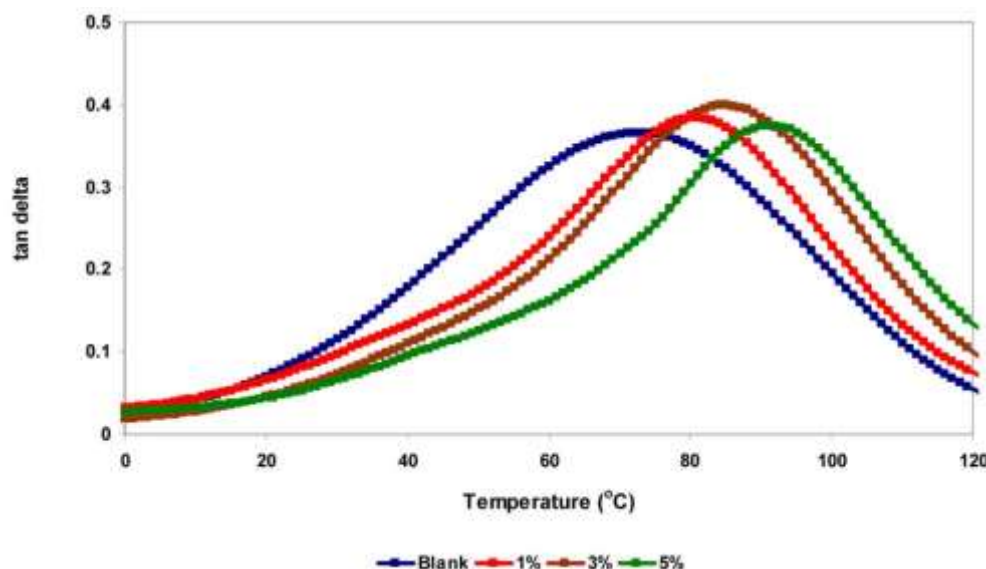
Figure 8. Diagram of tan δ for epoxy acrylate/polyaniline nanocomposites with different percentages of polyaniline.

4. Conclusion

The results obtained from the spectroscopy analysis have effectively confirmed the successful synthesis of the UV-cured epoxy acrylate and also polyaniline resins. The obtained images of scanning electron microscopy (SEM) have provided visual evidence of the appropriate distribution of polyaniline particles within the epoxy matrix. Upon increasing the weight percentage of polyaniline from 3% by weight to 5% by weight, a notable improvement in electrical conductivity has been detected. The differential thermal analysis (DTG) diagrams have showcased the existence of three distinct stages of weight loss, each corresponding to the presence of moisture and non-reacted materials, polyaniline, and epoxy acrylate, respectively. The thermal stability of the coating showed a great increase with the increase of polyaniline from 1% to 3%, and for the 5% sample, this increase was even greater. The elastic modulus decreased with the rise of polyaniline in the epoxy acrylate matrix and the loss modulus decreased at low temperatures and

increased in warmer conditions. Also, the glass transition temperature of the sample without polyaniline, which was increased to 93°C in the sample containing 5% polyaniline.

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