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Synthesis and Investigation on Viscoelastic Properties of Urethane Acrylate-Polyaniline

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ABSTRACT

The aim of this research is to investigate the viscoelastic behavior and determination of cross-link density of thin conductive coatings of urethane acrylate-polyaniline. For this purpose, polyaniline and urethane acrylate resin were synthesized. Nanocomposites with different percentages of polyaniline (1, 3, and 5%) were prepared and cured by ultraviolet irradiation. The viscoelastic properties of prepared thin films were evaluated by dynamic mechanical thermal analysis under stresses with a certain frequency at different temperatures. Also, cross-link density of nanocomposites was measured after ultraviolet curing. The results indicated that the glass transition temperature decreases and the cross-link density increases with the amount of polyaniline. Prog. Color Colorants Coat. 11 (2018), 241-252© Institute for Color Science and Technology.

1. Introduction

In recent years, nanocomposites containing conductive polymers have attracted a great deal of attention owing to various applications including solar cells, antielectricity coatings, anticorrosion coatings, and other similar purposes [1, 2]. Different usages of polymer nanocomposites have caused greater attention to their physical and mechanical properties as well as viscoelastic behavior [3, 4].

Polyaniline is a polymer which is intrinsically conductive and has a conjugate system, making a wide π network. Indeed, the source of conductivity of this polymer is the movement of electrons within the framework of π electrons. Doping elevates the conduction surface. In this process, a receiver or donating molecule is added to the couple system [5, 6]. Four important factors including molecular weight, crystalline percentage and inter-chain separation, level of oxidation and molecular arrangement, doping percentage and the type of doper may influence the electrical conductivity of polyaniline [7, 8].

Various tests are used to investigate the behavior of polymer compounds. To examine the viscoelastic behavior, three tests including creep test, stress relaxation and dynamic mechanical thermal test are used [9]. Dynamic mechanical thermal analysis (DMTA) is a test that is used for investigating the viscoelastic flow processes in polymers. This test allows for determining the properties and molecular movements of a certain substance within a range of temperatures [10]. In this test, a sine load is applied to a sample with a certain frequency within a specific range of temperature and time, and the sample's responses are recorded. This test has applications for measuring glass transition temperature, and alpha and beta relaxations [11, 12].

Loss modulus, storage modulus and tangent delta $(\tan \delta)$ are among the most important results which can be achieved by DMA test. Dynamic loss modulus (E') represents the viscosity of materials. Storage modulus (E') refers to Young modulus and determines the extent of rigidity of a material and tolerability of composite materials [13]. Mechanical damping parameter or tand is defined as $\tan \delta = E''/E'$, which is a dimensionless number [11, 13]. One of the applications of $tan\delta$ is to determine the glass transition temperature (T_{g}) . This temperature is one of the most important thermal properties for designing polymer composite materials [14]. Another application of this test is the investigation of cross-link density of polymer structures, which is performed using storage modulus and experimental relations [15-17].

In recent years, UV curing materials have attracted great interest for its eco-friendly, easy-to-process, high-speed curing of coatings and low energy consumption. UV curing resins are used in clear coating [18, 19], hybrid coatings [20], additives used in UV curing systems such as cross-link agents [21], and many other applications [22, 23].

In this research, conductive and fast cure coating is prepared. Fast cure is due to the use of UV-curable oligomer of urethane acrylate and the electrical conductivity of the polymer is related to the polyaniline. This UV-curable composite makes it possible to use it in solar cells, anticorrosion coatings, conductive adhesives, and those that require electrical conductivity. The viscoelastic behavior of conductive nanocomposites of polyaniline in UV curable urethane acrylate matrix is studied using dynamic mechanical analysis. Other properties including glass transition temperature, storage and loss modulus, and cross-link density are also analyzed.

2. Experimental

2.1. Materials

Aniline, ammonium persulfate (as the oxidant), HCl 37% (as the doper), isophorone diisocyanate (IPDI), acetone, triethylamine (TEA) as the co-initiator, 2-hydroxyethyl methacrylate (HEMA), dibutyltin dilaurate (DBTDL) catalyst and benzophenone (as the initiator) were all purchased from Merck Co. (Germany). Polyethylene glycol 400 (PEG400) and trimethy-lolpropane triacrylate (TMPTA) were purchased from Sinchem (South Korea) and Sigma Aldrich Company

(USA), respectively. Aniline was double distilled before using and other materials were used without any purification.

2.2. Synthesis of polyaniline (PANI)

Polymerization of polyaniline was carried out according to our previous work [24]. A total of 25 mL fresh distilled aniline and 625 mL HCl (2 M) was poured into 2 liters reaction container and then placed in ice water bath. Then, 250 mL ammonium per sulfate 0.25 M was added drop wise to the solution under continuous stirring. After adding a considerable amount of oxidant, the solution's color changed, indicating the onset of the polymerization reaction. Following complete addition of the oxidant, the mixture under stirring was remained overnight to complete the reaction. Then, the resulting polymer was filtered and washed several times with distilled water. The obtained polyaniline was dried for 12 h in vacuum oven at 50 °C.

2.3. Preparation of urethane acrylate (UA)

In order to prepare urethane acrylate, polyethylene glycol 400 (PEG400) and isophorone diisocyanate (IPDI) were used with 2:1 molar ratio. 0.118 mol (26.45 g) isophorone diisocyanate, 0.058 mol (23.55 g) PEG400, 5 mL acetone and 0.2 mL dibutyltin dilaurate were poured into the reaction container equipped with condenser, thermometer and nitrogen inlet. This mixture was stirred at 45 °C for 2 h by magnetic stirrer. Then. 0.118 mol (15.36 g) 2-hydroxyethyl methacrylate (HEMA) was added to the reactor, stirred for another 2-3 h. Synthesized urethane acrylate was placed in vacuum oven for 24 h at ambient temperature for removing the solvents. Subsequently, 16.85 g trimethylolpropane triacrylate (TMPTA) diluent (i.e. 33.7 wt% of the product) was added. Figure 1 represents the schematic synthesis of urethane acrylate.

2.4. Preparation of nanocomposite

For preparing the nanocomposites, 10 g urethane acrylate was mixed with different weight percentages of polyaniline (1, 3, and 5%), benzophenone (4 wt%) and triethylamine (4 wt%).

At first, polyaniline was mixed with 2 mL acetone and dispersed by ultrasound for 30 min. Then, urethane acrylate was added and stirred for 45 min in order to obtain a homogeneous mixture. In the next step, benzophenone was added and stirred for 5 min

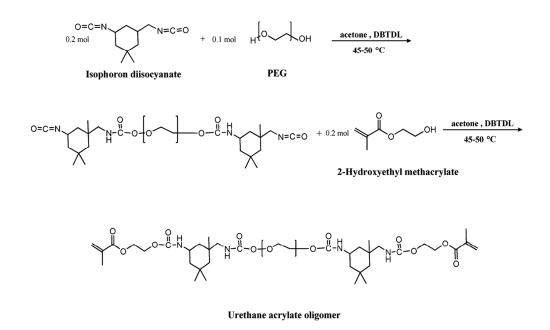


Figure 1: The schema of synthesis of urethane acrylate resin.

and at last, TEA was added and the mixture was completely mixed. The resulting mixture was placed in vacuum oven for removing the acetone. Nanocomposite films were prepared by applicator with a film thickness of 120 micron on clean glass plates. The prepared samples were cured under ultraviolet irradiation for 2 min.

2.5. Characterization

Ultrasound device Hielscher UP400s (Germany) for dispersing nanoparticles in the polymer matrix, vacuum oven WiseVen WOV-30-60 cmHg (South Korea) for drying and solvent removal, film applicator Neurtec Co. (Spain) for preparing the nanocomposite films and ultraviolet curing 1 kW, Hg lamp 80 W/cm² (China) for curing of nanocomposites were used.

Investigation of the morphology of the nanocomposites was performed by scanning electron microscopy (SEM) LEO1455VP (England). The viscoelastic behavior of nanocomposites was studied by Netzsch DMA 242C (Germany) with different frequencies (0.1, 0.5, 1, 5, 16.6 and 25 Hz.) at

temperature range of -150 to +150 $^{\circ}\mathrm{C}$ and heating range of 10 $^{\circ}\mathrm{C/min}.$

3. Results and Discussion

3.1. Morphology

In order to investigate the status of dispersion of nanoparticles in polymer matrix, scanning electron microscopy (SEM) was used. Figure 2 represents the microscopic images of the nanocomposites prepared with different weight percentages of polyaniline nanoparticles in the urethane acrylate matrix.

SEM images in Figure 2 indicate that aggregation of polyaniline increases with increasing the polyaniline nanoparticles in the urethane acrylate matrix. The polymer matrix containing more than 3 wt% polyaniline shows heterogeneity and agglomeration. So, the nanocomposite containing 3 wt% nanoparticles has better homogeneity and dispersion in terms of the others. These results were in line with the results of other studies which the suitable dispersion was at 3 wt% [11].

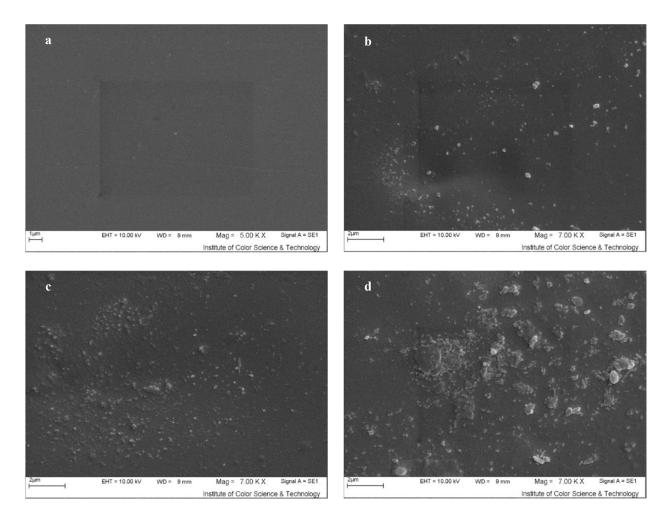


Figure 2: SEM images of a) pure acrylate urethane (Blank), b) urethane acrylate + 1% polyaniline, c) urethane acrylate + 3% polyaniline, and d) urethane acrylate + 5% polyaniline.

3.2. Investigation of the viscoelastic behavior

3.2.1. Investigation of glass transition temperature

Glass transition temperature (T_g) is a phase alteration which occurs in solids such as glasses, polymers and some metals. This temperature is defined as a temperature at which an amorphous material undergoes a physical change from rigid and fragile to flexible and rubber-like conditions. Typically, E' peak or tan δ is a criterion for T_g, though, in many researches, tan δ is used, whose peak is some degrees above that of E". Tan δ mainly represents the middle point of the transition region of E' logarithmic curve reduction, whereas the loss modulus peak refers to initial drop from glass state to transition region [12].

The tan δ peak is related to the glass transition temperature (T_g) of the polymer and represents the limits of movement of polymer chain in the nanocomposite [11]. The highest part of tan δ peak

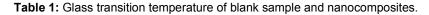
specifies the amount of T_g . Figure 3 demonstrates the changes in tan δ or, in other words, changes in glass transition temperature of the nanocomposites.

Table 1 presents the values of glass transition temperature. According to Figure 3 and data in Table 1, the glass transition temperature decreases with increasing the amount of polyaniline in the urethane acrylate matrix. This is probably due to the interaction between polyaniline and the polymer matrix. Interactions with the polymer matrix cause development of friction forces among the chains, limiting the molecular mobility and increasing the glass transition temperature [25].

3.2.2. Investigation of the storage or elastic modulus

Storage or elastic modulus (E') is the elastic or solid like characteristic of the material and refers to rigidity

in a material. In Hook's law, this modulus is the ratio of stress to strain and the ability to store energy for a certain material. The higher the E' value, the greater is the elastic property in the material [9, 10, 12]. Figure 4 indicates the changes in the elastic modulus in terms of temperature for all nanocomposites.



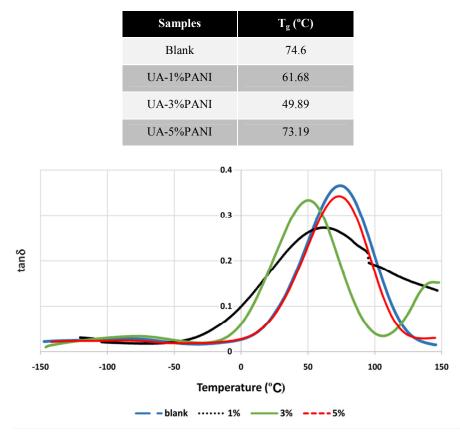


Figure 3: Variations of tano in terms of temperature at frequency of 1 Hz.

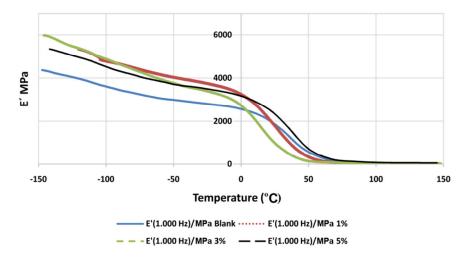


Figure 4: Variations of storage modulus (E') at 1 Hz.

The beginning point of the reduction in the elastic modulus is one of the points that can be considered as glass transition temperature [10]. The diagram in Figure 4 demonstrates that with the increase in the amount of polyaniline nanoparticles in the urethane acrylate matrix, the elastic modulus has increased and the beginning point of the reduction in the elastic modulus is transferred to a higher temperature, suggesting elevated glass transition temperature.

The increase in the elastic modulus can be attributed to the adhesion between the filler and the polymeric matrix. One of the applications of Young modulus is determining the adhesion between the filler and polymeric matrix. Indeed, if the elastic modulus does not grow by increasing the filler amount, this means that the filler has a weak adhesion to the polymeric matrix, causing aggregation or accumulation. In SEM images, it is observed that the sample with 3% nanoparticle has better homogeneity, but by increasing the nanoparticles to 5%, the accumulation of the filler becomes greater and the elastic modulus also increases to some extent. This increase in elastic modulus can suggest elevated adhesion between polyaniline nanoparticles and urethane acrylate, depending on polymer type (amino, mercapto, epoxy or vinyl are the most common functional groups which react with polymer) [25].

Furthermore, a descending trend was observed in the storage modulus for all samples. This can be due to the fact that with the increase in the temperature, the free space in the polymer chain increases and the local movement of chains converts to a global movement. Therefore, the storage modulus curve changes from a smooth and uniform state to a sloped curve for, which is descending.

3.2.3. Investigation of the changes in the loss modulus

Loss modulus represents the energy wasted in a viscoelastic sample as heat and it is the viscosity characteristic with a liquid-like nature of the material [9, 10, 12]. This modulus is associated with internal friction of a substance and is sensitive to their molecular movements of relaxation processes, morphology, and heterogeneities [10]. Figure 5 demonstrates the changes in the loss modulus in the synthesized nanocomposites.

The diagram in Figure 5 indicates that with the increase in temperature, loss modulus has declined in the samples and changed into lower values than the blank sample. Table 2 provides the loss peak values and the loss temperature. In this table, it is evident that with the increase in temperature, the loss peak has changed into lower values. Loss modulus is the characteristic of the viscous state of a material. In scientific documents, elevation of loss modulus in response to increase of filler content is considered to be associated with the friction among the filler particles and the polymer matrix molecules [10].

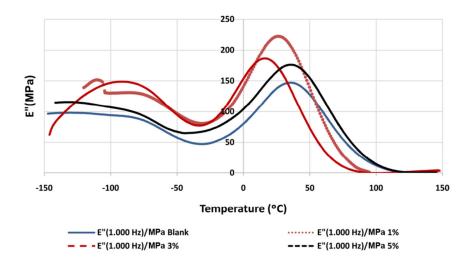


Figure 5: The loss modulus (E") in terms of temperature at the frequency of 1 Hz.

| Samples | Loss peak (MPa) | Loss peak temperature (°C) |
|-----------|--------------------|-------------------------------|
| Blank | 147.1046 | 35.62534 |
| UA-1%PANI | 222.4802 | 26.68633 |
| UA-3%PANI | 186.0094 | 15.89467 |
| UA-5%PANI | 175.9140 | 35.19934 |

Table 2: Variations of loss modulus peak of the nanocomposites.

With increasing the filler in the polymer matrix, the friction among the molecules increases, this can shift the loss modulus peak to a higher temperature. In this research, the loss peak experienced a reduction, which can be due to the diminished friction among polyaniline molecules and the urethane acrylate matrix. This is because, by the growth of temperature, the mobility in the polymer chain increases due to development of free volume. Table 2 presents the values of loss peak in the second test, in which the descending trend of the values is evident.

3.2.4. Variations in the activation energy using Arrhenius equation

Activation energy is one of the important parameters in polymers. Determination of activation energy contributes to determine the glass transition temperature [26], as at the glass transition temperature, the tan δ diagram reaches its maximum. As a result, at this point, the level of activation energy should be maximum and thus the maximum point of activation energy represents the glass transition temperature. Arrhenius equation is used (Eq. 1) to calculate the activation energy [16, 17, 27].

$$\log(a_T) = \frac{E_a}{R} \left(\frac{1}{T} - \frac{1}{T_0} \right)$$
(1)

where a_T is the horizontal shift factor, R is the global constant of gases, and E_a is the activation energy. Calculation of activation energy can be done by plotting the diagram. By rewriting the equation 1 as Eq. 2 and plotting the changes of natural logarithm of frequency in terms of temperature, through calculation of the obtained slope of the line, the activation energy value can be achieved [27].

$$\Delta H = -R \frac{d(\ln f)}{d(\frac{1}{Tg})}$$
(2)

In equation 2, activation energy value can be calculated through multiplying the slope obtained from plotting the diagram of lnf versus T by the global constant of gases. Figure 6 indicates the line equation and its slope value for all of the nanocomposites. Table 3 summarizes the activation energy values obtained for urethane acrylate and the prepared nanocomposites.

As can be seen in Table 3, with the increase in the weight percentage of polyaniline nanoparticles in urethane acrylate matrix, glass transition temperature increases. These results are congruent with the results obtained from calculation of glass transition temperature obtained by interpolating the diagrams, suggesting the accuracy of the glass transition temperatures obtained from the diagrams. This is because the point of glass transition temperature in the diagram of tanð versus temperature is located where the diagram reaches its peak. At this peak, the activation energy reaches its maximum. In other words, at the glass transition temperature, the activation energy value is a maximum.

3.2.5. Effects of frequency on elastic modulus, loss modulus and glass transition temperature

The frequency is one of the factors influencing molecular movements and glass transition temperature. Glass transition temperature is accompanied by important molecular relaxation processes and segmental movements in the polymer chain. The rate of these segmental movements depends on temperature. By increasing the frequency, the relaxation processes in the glass transition region would not be able to keep their previous state in response to the stresses exerted to the sample. In this way, the polymer moves towards rigidity. Therefore, segmental movements occur at higher temperatures and thus the glass transition temperature grows [9].

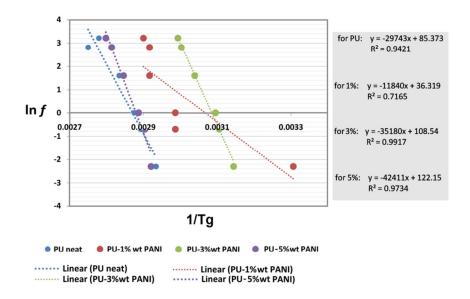


Figure 6: Variations of frequency versus temperature for calculating activation energy at the glass transition temperature.

| Table 3: The activation | energy value in the | nanocomposites | (in kJ). |
|-------------------------|---------------------|----------------|----------|
| | | | |

| Samples | Activation energy (kJ) | |
|-----------|------------------------|--|
| Blank | 247.28 | |
| UA-1%PANI | 98.4 | |
| UA-3%PANI | 292.4 | |
| UA-5%PANI | 352.6 | |

Arrhenius relation indicates mathematical relationship between frequency and temperature and it is used for investigating the effect of frequency on the response of samples in DMTA tests, which is expressed as equation 3 [17].

$$f = A e^{\frac{-Ea}{RT}}$$
(3)

In equation 3, Ea represents activation energy and R is the constant of gases. As can be seen from this relation, frequency and glass transition temperature are directly related [17, 27]. Figures 7 and 8 well indicate this concept in the conducted tests, where with the increase in the frequency, loss modulus and glass transition temperature have increased.

As can be seen in Figures 7 and 8, it is evident that both the loss peak and $tan\delta$ peak increase with frequency. The tand peak represents the limit of molecular mobility, at which some movements occur in the main chain of the polymer and after that the polymer moves towards rubbery. When the tangent peak emerges at a higher frequency, this means that the limiting effect of filler particles in the nanocomposite has been very important and the mobility of the polymer chain has severely been limited [11]. Due to the effects of frequency on molecular movements, DMA test is conducted at the standard frequency of 1 Hz, though conductance of this test at different frequencies is a suitable method for investigating viscoelastic properties in designing damping vibration devices [9, 28].

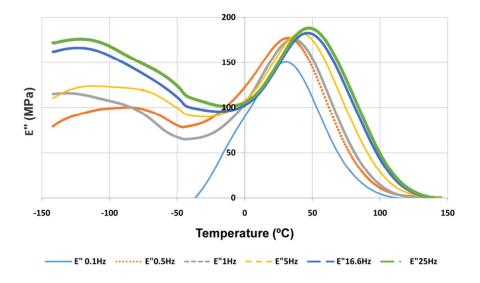


Figure 7: Variations of loss modulus with temperature at various frequencies for the 5% nanocomposite.

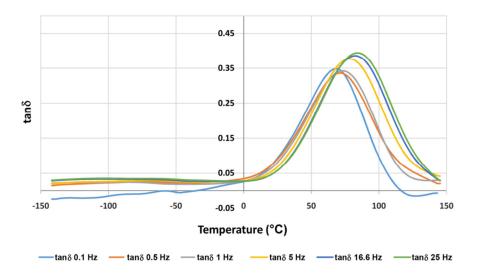


Figure 8: Variations of tano in terms of temperature at various frequencies for 5% nanocomposite.

3.2.6. Cross-link density

Cross-linking is one of the phenomena that occur during the curing of polymers. During cross-linking, a kind of cross-link is developed among polymer chains, causing various cross-linked to structures in them and today identification of these the structures has become of interest to many researchers [15, 29]. To our knowledge, there is no direct method for calculating the cross-linking degree. Typically, with the increase in the cross-linking density, molecular movement in the material diminishes, referring to elevated glass transition temperature [29]. Considering cross-linking, a number of experimental relations have been presented. One of them which have been referred to in many literatures is equation 4 [29, 30].

$$v_e = \frac{E'}{3RT} \tag{4}$$

where v_e represents the molar concentration of cross-linking in terms of mol/m³, E' is the elastic modulus in terms of Pascal, T is temperature in terms of Kelvin, and R is the constant of gases whose value is 8.31 J/K here. This equation is valid when the elastic modulus lies within the range of 2×10^6 and 2×10^8 Pa.

The obtained results are provided in Table 4.

Furthermore, Nielsen equation (Eq. 5) is applicable for estimating cross-linked polymer structures. This equation relates glass transition temperature to the molecular weight of the polymer chains during crosslinking [30, 31].

$$Tg = Tg_0 + \frac{3.9 \times 10^4}{Mc}$$
(5)

where T_g denotes the glass transition temperature of the cross-linked polymer and T_{g0} shows the glass transition temperature within non-cross-linked range and \overline{Mc} denotes the mean molecular weight of the polymer chains among the links in terms of g/mol. Another equation that exists for cross-linked polymers is Eq. 6 [30, 31].

$$Tg = Tg_0 + \frac{788}{nc} \tag{6}$$

where n_c represents the average number of atoms in the main chain of the polymer in the cross-linked network. In a research conducted on a cross-linked acrylate film (cured), T_{g0} value has been calculated through mathematical extrapolation [32]. Here, this value has also been estimated to be 58.808 °C using this mathematical method [28]. Results at the frequency of 1 Hz are presented in Table 5.

As can be seen in Table 5, the values of Mc and n_c decrease. Typically, Mc is a criterion for cross-linking and represents the cross-linking degree in terms of 1/Mc [30, 31]. The reduction in the value of Mc or elevation of 1/Mc suggests elevated degree of cross-linking. Based on the results obtained from Table 5 regarding the values obtained for Mc, it can be stated that the increase in polyaniline in the polymer matrix rises cross-linking in this nanocomposite. Figure 6 demonstrates the extent of cross-linking in terms of the weight percentage of polyaniline in the urethane acrylate matrix.

Table 4: Variations of cross-linking density in the nanocomposites at the frequency of 1 Hz.

| Samples | T _g (°C) | E'(MPa) | $v_e (mol/m^3)$ |
|-----------|---------------------|----------|-----------------|
| Blank | 74.62534 | 115.2751 | 13295.79913 |
| UA-1%PANI | 61.6863 | 124.9125 | 14964.11674 |
| UA-3%PANI | 44.89464 | 142.768 | 18006.1356 |
| UA-5%PANI | 73.19934 | 164.0384 | 18998.04367 |

Table 5: The results of Neilsen equation.

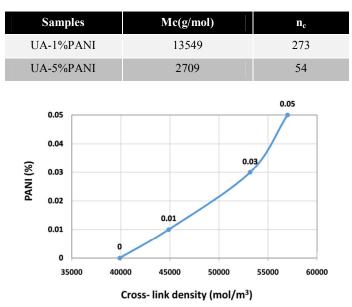


Figure 9: Variations of cross-link density in terms of percentage of polyaniline.

Figure 9 demonstrates that with increasing the percentage of polyaniline in the urethane acrylate matrix, cross-link density increases and causes less mobility of polymer chains, hence increasing the T_g of polymer.

4. Conclusion

In this research, the viscoelastic properties of polyaniline conductive nanoparticles in urethane matrix were investigated by dynamic mechanical thermal analysis (DMTA) test. The results indicated that the glass transition temperature increases with the amount of polyaniline nanoparticles. This is due to the fact that with the increase in amount of polyaniline, some links are developed between the nanoparticles and polymer matrix. Aggregation, which is evident in SEM micrographs, results in reduced polymer chain mobility. Furthermore, the values of elastic modulus have increased to some extent but the viscous modulus has decreased. However, the value of elastic modulus has been always greater than that of loss modulus, suggesting the elastic property of the sample. This is related to the increased molecular aggregation of polyaniline particles in the polymeric matrix, which causes rigidity of the polymer chain and makes its movement more difficult. Also, the increase in the elastic modulus represents elevated adhesion between

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the polyaniline and the polymer matrix. Here, with the increase in the amount of nanoparticle in the polymer matrix, the elastic modulus has slightly increased, suggesting that adhesion has enhanced between the nanoparticle and the polymer chains. According to Arrhenius equation, temperature has a direct relationship with frequency and the elevation of frequency also increases the glass transition temperature. This arises from the limited mobility of the polymer chain due to the presence of polyaniline in the polymer matrix. The amine groups in the polyaniline are one of the factors of interaction between the polyaniline and the polymer matrix and diminished mobility of the polymer chains. Calculation of activation energy is a good method for investigating the glass transition temperature calculated in the diagrams. At glass transition temperature, the value of activation energy is a maximum and it indicates a good correspondence such that by increasing the glass transition temperature, activation energy also increases. Furthermore, cross-linking calculations revealed that the extent of cross-linking increases with the addition of polyaniline to urethane acrylate. Cross-linking causes development of cross-links among the polymer networks, thereby causing further reduction in the mobility of the polymer chains and transferring glass transition temperature to higher temperatures.

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