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Synthesis, Characterization, Optical and Thermal Properties of P(NVC-co-BZMA) Copolymer and Its ZnO Composites

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Keywords

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Abstract

Recent studies have paid particular attention to polymer-nanoparticle composite materials considering they have a number of interesting properties, which include optical, thermal, electrical, and others (Reyna-Gonzalez et al., 2009; Yakuphanoglu et al., 2010). In this study, a free radical polymerization process was used to create a copolymer of N-vinylcarbazole (NVC) and benzyl methacrylate (BZMA) at 25–75 mol% each. The procedure was conducted at 70°C with azobisisobutyronitrile (AIBN) acting as the initiator. Nano zinc oxide powders were then added to composites at three different ratios of 5%, 10%, and 15% weight of the copolymer. In order to better understand the structures of the P(NVC-co-BZMA) and its composites, FT-IR, ^{1}H NMR, and UV spectroscopic techniques were also implemented. The optical characteristics of both the pure copolymer and its composites were examined. In the visible region, the composite containing 15% nano ZnO had the highest optical absorbance value. Additionally, the thermal behaviours of the composites and copolymers were analysed.

Cite

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1. INTRODUCTION

The presence of two monomers with different properties in a polymer chain with different compositions can give the polymer new properties (Saçak, 2012). Copolymerization is a method that can be used for this purpose and is of great technological importance. Furthermore, the structures obtained by adding nano- or macro-sized reinforcing materials to polymers and called polymer composites significantly change the properties of polymer materials.

NVC and BZMA monomers are among the polymers studied in copolymerization reactions in terms of some the properties that they exhibit. The high softening temperature of N-vinylcarbazole, an aromatic vinyl monomer, has caused it to become the subject of copolymerization studies with different monomers. One of the aims of these studies has been to obtain polymers with a lower softening temperature than P(NVC) (Isusi et al., 2006). Moreover, NVC has attracted attention due to its optical applications and is considered worthy of research in this field. There have also been composite studies with ZnO to improve the properties of P(NVC) (Ghosh & Biswas, 2009). Ghosh et al. (2010) studied the dielectric properties of P(NVC), a conducting polymer, by preparing its composites with acetylene black and ZnO separately and found that the nanocomposite of poly(N-vinylcarbazole) with acetylene black exhibited a negative dielectric constant at high frequency. Goumri et al. (2020) synthesized composites of P(NVC) with graphene oxide and emphasized that the electrical conductivity can be improved depending on the varying graphene oxide ratio. Benzyl Methacrylate (BZMA) is a methacrylic monomer containing the phenyl group in its structure. Although it has many applications (Aqel et al., 2013; Li et al., 2014), it has generally been used in

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copolymerization studies to obtain thermal functionality (Ueno, 2018). For example, Liu et al. (2022) synthesized a series of copolymers with different BZMA and MMA compositions as microspheres and emphasized that some properties of the copolymer such as thermal stability and glass transition temperature (Tg) can be changed depending on the monomer compositions. There are also composite studies on BZMA in the literature. Demirelli et al. (2022a) prepared composites of BZMA with lactone end groups with graphene and reported that the composite material's thermal stability saw an increase that followed that of the amount of graphene doped, but the Tg value decreased.

Thermal degradation kinetic data of polymers is an important parameter to understand the degradation process of the polymer. Thermogravimetric analysis (TGA) is one of the most often used methods of analysis in the examination of the kinetics of polymer thermal degradation and it entails evaluating the activation energy of the thermal degradation of polymers. All kinetic studies are based on the assumption that the isothermal rate conversion dα/dt is a linear function of k(T), which is a temperature-dependent rate constant, and f(α), which is a function of conversion independent of temperature. This is written as follows:

$$\frac{d\alpha}{\mathrm{d}t} = k(T)f(\alpha) \tag{1}$$

The activation energy is normally pinpointed through attaching kinetic data to various reaction models, yielding the Arrhenius parameters:

$$k(T) = \mathrm{A}\exp\left(-\frac{\mathrm{Ea}}{RT}\right) \tag{2}$$

Here, A is a pre-exponential factor assumed to be independent of temperature, E_a represents activation energy, T is absolute temperature, and R is the universal gas constant (R = 8.314 J/mol K). When these are combined, the following equation can be written:

$$\frac{\mathrm{d}\alpha}{\mathrm{dt}} = \mathrm{A}\exp\left(-\frac{\mathrm{Ea}}{\mathrm{RT}}\right)f(\alpha) \tag{3}$$

For non-isothermal measurements at a constant heating rate of $\beta = \frac{dT}{dt}$ equation (3) transforms to the following:

$$\beta\left(\frac{\mathrm{d}\alpha}{\mathrm{dT}}\right) = \mathrm{A}\exp\left(-\frac{\mathrm{Ea}}{\mathrm{RT}}\right)f(\alpha) \tag{4}$$

Activation energy E_a may be determined by a variety of techniques and, in particular, the Flynn-Wall-Ozawa (FWO) and Kissinger-Akahira-Sunose (KAS) models are frequently utilized in the calculation of the activation energy of polymer thermal degradation (Barim, 2021; Demirelli et al., 2022b). Both are integral methods that are independent of the reaction order. In the Flynn-Wall-Ozawa method, parallel lines are attained by plotting *Log β* against $1/T(K^{-1})$ various rates of heating. By calculating the slope of these lines, the activation energy (Ea) is achieved. The FWO method uses the following equation 5 (Ozawa, 1965; Flynn & Wall, 1966):

$$log(\beta) = \log\left[\frac{\mathrm{AEa}}{\mathrm{g}(a)R}\right] - 2.315 - \frac{0.457\ \mathrm{Ea}}{\mathrm{RT}} \tag{5}$$

In the equation above, g(α) is an unknown function of conversion. According to this equation, the activation energy $(-0.457\ Ea/RT)$ is calculated using the slope of the *Log β* - $1/T(K^{-1})$ graph.

When implementing the KAS method to uncover the activation energy, $\ln(\beta/T^2)$ is plotted against $1/T(K^{-1})$. The Ea value is calculated from the slope of the linear line obtained from this graph. The equation of the KAS method is defined as follows (Kissinger, 1957):

$$\ln\left(\frac{\beta}{T^2}\right) = \ln\left(\frac{\mathrm{AR}}{\mathrm{g}(\alpha)\mathrm{Ea}}\right) - \frac{\mathrm{Ea}}{\mathrm{RT}} \tag{6}$$

The primary purpose of the present study was to synthesize, characterize, and make composites of a copolymer not encountered in the literature. Following this, it was to reveal the thermal degradation parameters, activation energies (with FWO and KAS method), and optical properties of both copolymer and composites. For this purpose, the P(NVC-co-BZMA) copolymer was synthesized, its structure was elucidated, and the composites of this copolymer with 5, 10 and 15 wt% nano ZnO were then prepared. Finally, the thermal and optical properties of copolymers and composites were investigated.

2. MATERIALS AND METHODS/EXPERIMENTAL METHODS

Benzyl methacrylate (BZMA) and N-vinylcarbazole (NVC) monomers were purchased commercially from Sigma-Aldrich. ZnO NPs a purity of 99.5% were obtained from Nanography Nanotechnology (Ankara, Turkey).

2.1. Preparation of P(NVC-co-BZMA) Copolymer

The P(NVC-co-BzMA) copolymer was synthesized by free radical polymerization method using 25 mol% NVC and 75 mol% BZMA monomers in the company of the AIBN initiator in 1,4-dioxane solvent submerged in a 70°C oil bath. Polymerization was performed for 12 hours. The copolymer was precipitated in ethyl alcohol solvent and this process was repeated 3 times. It was later dried in a vacuum oven at 40 °C for 24 hours. Structural characterization of the copolymer was performed by the spectroscopic methods of FT-IR and ^{1}H NMR. Thermal characterization was also carried out with a TGA-DTA system. UV spectra were recorded and optical properties were analysed. The reaction scheme for the poly(NVC-co-BZMA) copolymer system is shown in Figure 1.

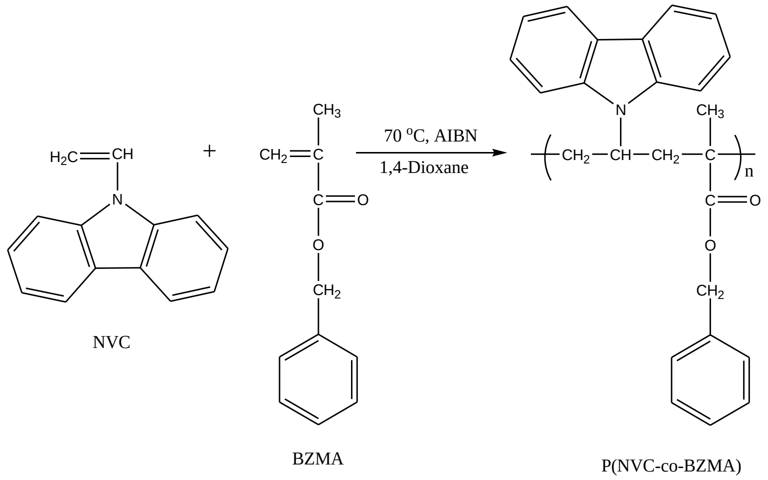

Figure. 1. *Synthesis scheme of the poly(NVC-co-BZMA) copolymer*

2.2. Preparation of P(NVC-co-BZMA) Copolymer Composites with Nano ZnO

Three composites of P(NVC-co-BZMA) copolymer containing 5, 10, and 15 wt% nano ZnO were prepared using the solvent casting method. Accordingly, certain amounts of P(NVC-co-BZMA) copolymers were dissolved in dichloromethane solvent in three separate beakers. Then 5, 10, and 15 wt% nano ZnO was weighed according to the amount of dissolved copolymer and dispersed in the dichloromethane solvent in three separate beakers for 2 h. Finally, the dispersed nano ZnO and copolymer solutions were mixed. The obtained nanocomposites were first mixed using an ultrasonic pen for two min and then the mixing process was continued with a magnetic stirrer overnight and the solvent of the composites was removed in a rotary evaporator. The composites were dried at a temperature of 40 °C inside a vacuum oven before determining the constant weight. Structural characterization of the composites was performed using the FT-IR spectroscopic method and thermal characterization by TGA-DTA dual system. UV spectra were taken and the optical properties were analyzed.

3. RESULTS AND DISCUSSIONS

3.1. FT-IR Spectroscopy

When the FT-IR spectrum of the P(NVC-co-BZMA) copolymer shown in Figure 2 was examined, the main peaks characterizing the copolymer were found as follows: peaks of aromatic C-H stretching vibrations between 2980–3048 cm^{-1}, peaks of aliphatic C-H stretching vibrations between 2840 and 2980 cm^{-1}, peak of ester carbonyl at 1723 cm^{-1}, peak of C=C stretching vibrations in the aromatic ring at 1450 cm^{-1}, peak of vinylidene at 1330 cm^{-1}, peaks of the (-C=O)-O-C group between 1156 and 1240 cm^{-1}, and peaks of C-H stretching vibrations of monosubstituted benzene at 695 cm^{-1}. The peak at 837 cm^{-1}, due to the nano ZnO, was below the peak at the same location in the composite structure and caused this peak to broaden (Haldar et al., 2011; Bashir et al., 2022).

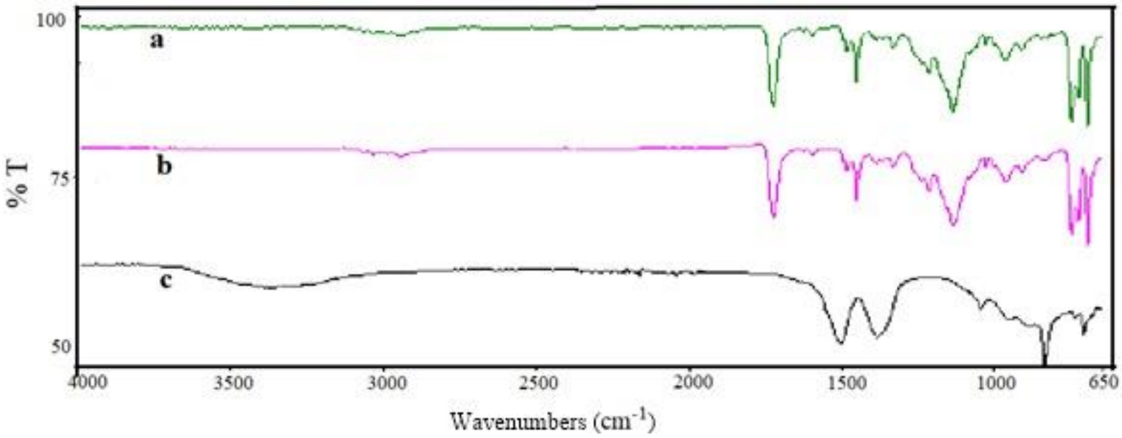

Figure. 2. *FT-IR spectra of a) P(NVC-co-BZMA), b) P(NVC-co-BZMA)/ZnO 15%, and c) nano ZnO*

3.2. ^{1}H-NMR Spectroscopy

The ^{1}H NMR spectrum of the copolymer which can be observed in Figure 3 was obtained using a Bruker 400 MHz AVIII 400 model NMR device in $CDCl_3$ solvent. In the ^{1}H NMR spectrum of the copolymer, the peaks between 0.5 and 2 ppm indicated CH_3 and CH_2 protons in the main chain of the polymer, the peak at 4.9 ppm indicated O-CH_2 protons belonging to the BZMA monomer, and the peaks between 6.8 and 7.9 ppm indicated protons originating from the aromatic ring in the NVC and BZMA monomers.

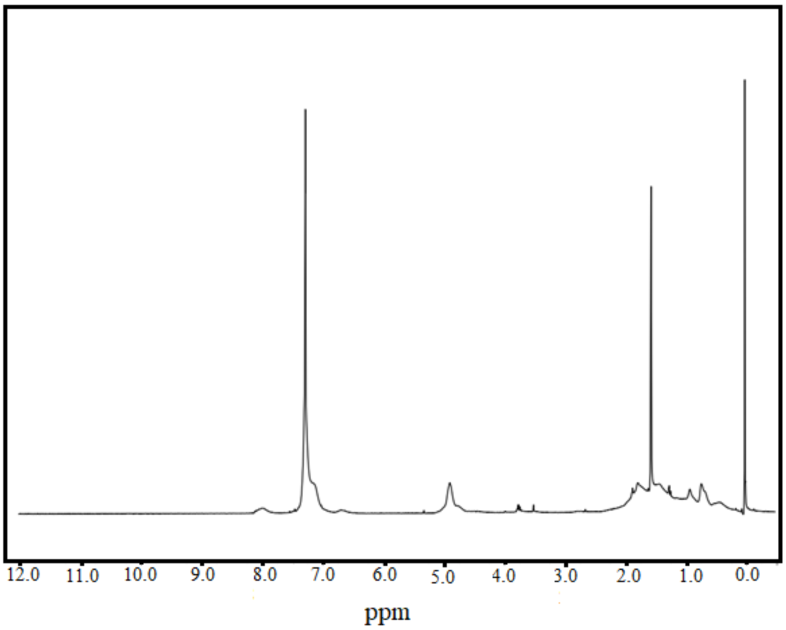

Figure. 3. *^{1}H NMR Spectrum of the P(NVC-co-BZMA)*

3.3. Thermal Analyses of the Copolymers and Composites

Determination of the copolymer and composite TGA curves required the use of a Perkin Elmer SII 7300 model TGA/DTA dual system. The thermal analysis curves of the copolymers and composites were obtained by heating them to 500 °C in aluminium vessels at a rate of 20 °C/min in a nitrogen gas atmosphere and are shown in Figure 4. Initial degradation temperatures, glass transition temperatures (Tg), temperatures corresponding to a given degradation, and Table 1 shows the waste calculations taken from TGA thermograms.

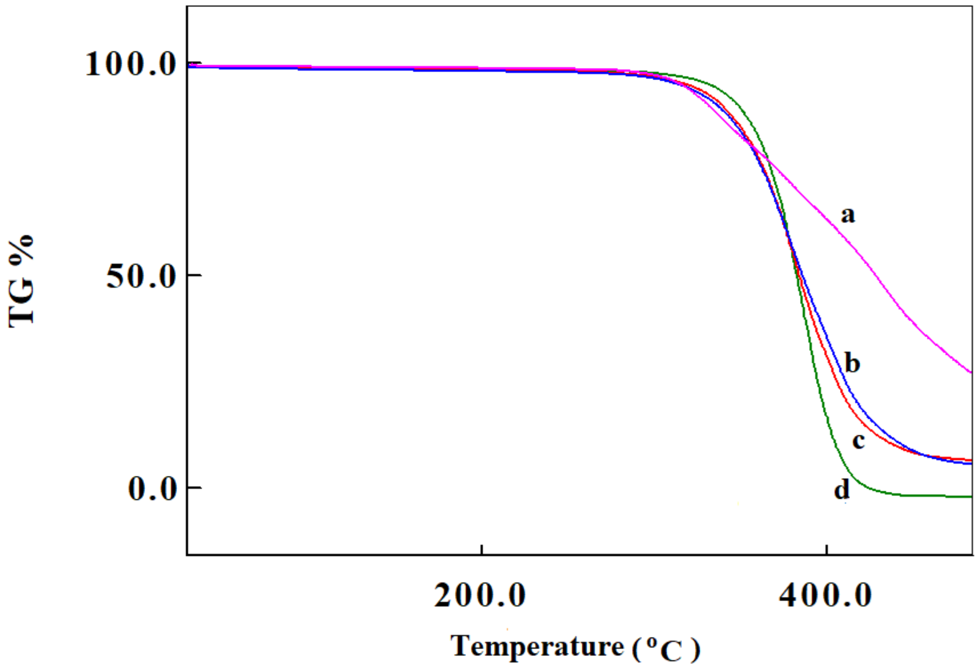

Figure. 4. *TGA curves of the copolymers and their composites; a) P(NVC-co-BZMA)/ZnO 15%, b) P(NVC-co-BZMA)/ZnO 10%, c) P(NVC-co-BZMA)/ZnO 5% and d) P(NVC-co-BZMA)*

Table 1. *The results of 20°C TGA for P(NVC-co-BZMA) and composite systems (Decomposition characteristics of copolymer and its composites with ZnO)*

Polymer / Composite	**Tg (°C)**	**IDT^a (°C)**	**Temperatures (°C) at certain % weight losses**					
			10	**30**	**50**	**70**	**90**	**% Residue at 500°C**
P(NVC-co-BZMA)	94	310	348.5	371	382	392	404.5	-
P(NVC-co-BZMA)/ZnO 5%	96	299	339	368.2	383.5	400	-	6.5
P(NVC-co-BZMA)/ ZnO 10%	98	300	336	367.7	385	405	443	5.5
P(NVC-co-BZMA)/ ZnO 15%	99	302	331.4	382.5	427	473.7	-	27

a: *Initial decomposition temperature*

3.3.1. Calculation of the Activation Energy of P(NVC-co-BZMA) and P(NVC-co-BZMA)/ZnO 10%

The thermal degradation kinetics of the P(NVC-co-BZMA) and P(NVC-co-BZMA)/ZnO 10% were examined by means of TGA thermograms. For this purpose, both the copolymer and composite samples were heated under nitrogen atmosphere from 30 °C to 500 °C; the rate was 10, 15, 20, and 25 °C/min for copolymer samples and 10, 15, 25, and 35 °C/min for composite samples. From the obtained thermograms, the degradation temperatures of the polymer with different transformations were determined. Using these data, the thermal degradation activation energies of the copolymer and composite samples were calculated according to the FWO and KAS methods. Thermal degradation activation energy plots of the P(NVC-co-

BZMA) copolymer calculated based on the FWO and KAS methods have been provided in Figure 5 and 6. The calculated values for activation energy according to these methods are shown in Table 2.

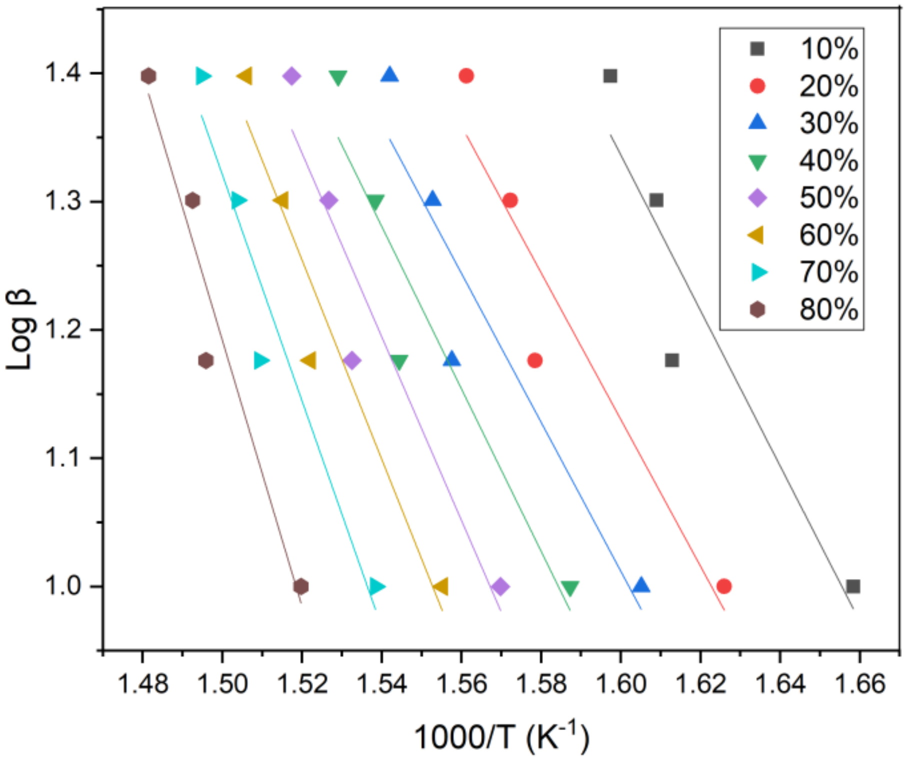

Figure. 5. *Determination of the activation energy of P(NVC-co-BZMA) calculated by FWO models*

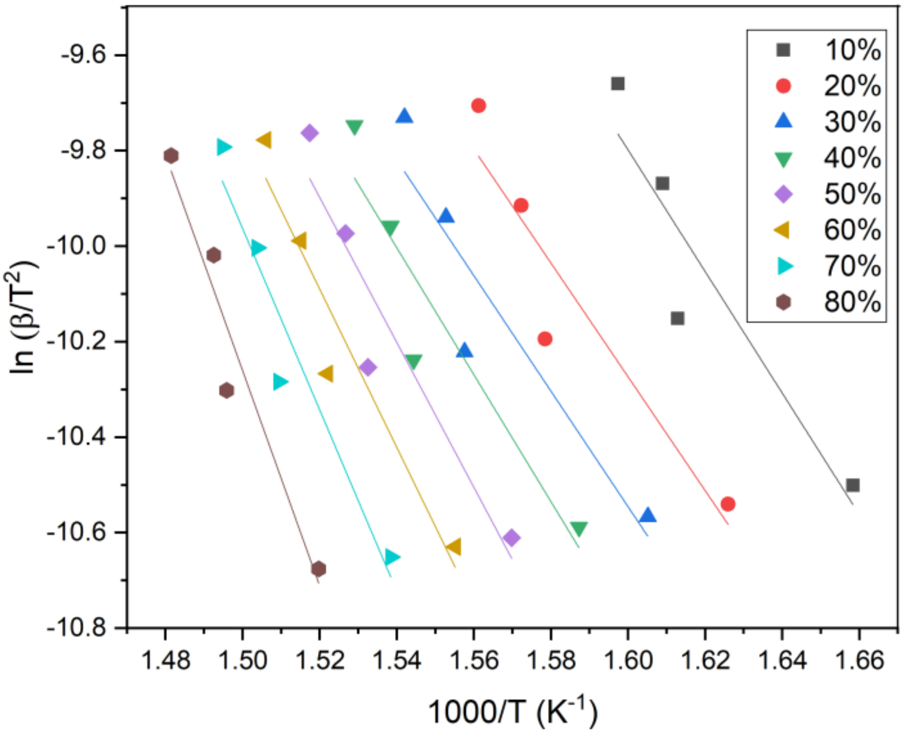

Figure. 6. *Determination of the activation energy of P(NVC-co-BZMA) calculated by KAS models*

Thermal degradation activation energy plots of the P(NVC-co-BZMA)/ZnO 10% composite calculated according to the FWO and KAS methods can be observed in Figure 7 and 8, while calculated values for activation energy according to both methods are highlighted in Table 3.

Table 2. *Activation energy values estimated by FWO and KAS Methods for P(NVC-co-BZMA)*

Flynn–Wall–Ozawa (FWO) Method		Kissinger–Akahira–Sunose (KAS) Method	
Conversion/α	**Ea (kJ/mol)**	**Conversion/α**	**Ea (kJ/mol)**
0.1	110.257	**0.1**	105.820
0.2	104.025	**0.2**	99.044
0.3	105.602	**0.3**	100.574
0.4	115.144	**0.4**	110.509
0.5	130.105	**0.5**	126.148
0.6	141.214	**0.6**	137.746
0.7	160.431	**0.7**	157.866
0.8	188.620	**0.8**	187.414
Average	**131.924**	**Average**	**128.140**

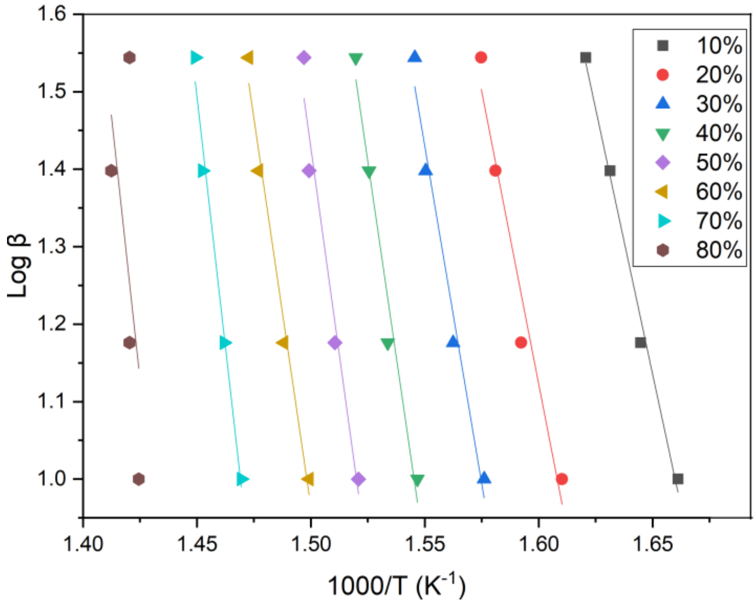

Figure. 7. *Determination of the activation energy of P(NVC-co-BZMA)/ZnO 10% calculated by FWO models*

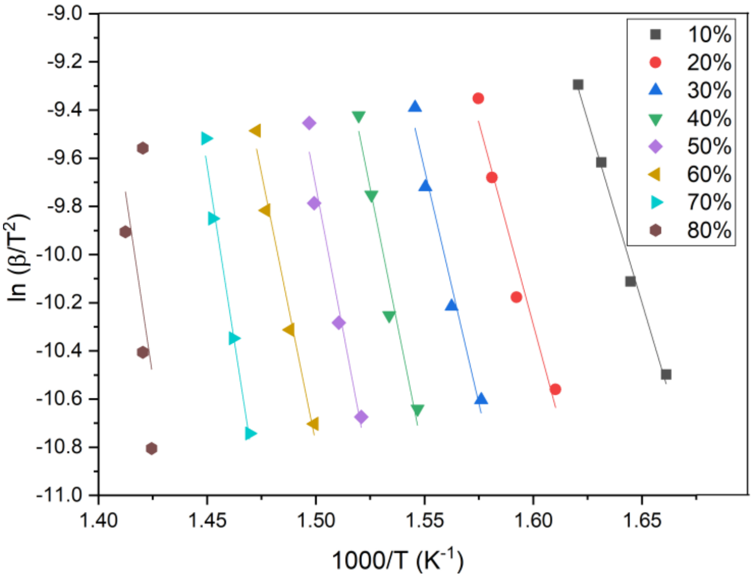

Figure. 8. *Determination of the activation energy of P(NVC-co-BZMA)/ ZnO 10% calculated by KAS models*

Table 3. *Activation energy values estimated by FWO and KAS Methods for P(NVC-co-BZMA)/ ZnO 10%*

Flynn–Wall–Ozawa (FWO) Method		**Kissinger–Akahira–Sunose (KAS) Method**	
Conversion/α	**Ea (kJ/mol)**	**Conversion/α**	**Ea (kJ/mol)**
0.1	249.183	**0.1**	242.080
0.2	274.653	**0.2**	268.580
0.3	317.096	**0.3**	313.015
0.4	367.162	**0.4**	361.518
0.5	388.447	**0.5**	383.725
0.6	364.633	**0.6**	360.500
0.7	470.459	**0.7**	470.658
0.8	492.927	**0.8**	493.982
Average	**365.573**	**Average**	**361.757**

3.4. Optical Properties

P(NVC-co-BZMA) films doped with 5, 10, 15 wt% ZnO were prepared using the sol gel drop coating method. UV measurements on the thin film samples were taken with a UV3600 SHIMADZU UV-VIS-NIR spectrophotometer. Transmittance and absorbance graphs were drawn using the data obtained from UV measurements. UV spectra of P(NVC-co-BZMA) copolymer and composites were examined. Results are shown in Figure 9 and 10.

The transmittance-wavelength spectra revealed that the undoped P(NVC-co-BZMA) copolymer had the highest optical transmittance in the visible region when compared to its nano ZnO-doped composites. In the same region, the P(NVC-co-BZMA)/ZnO 15% composite with the highest doping ratio was found to have the lowest optical transmittance.

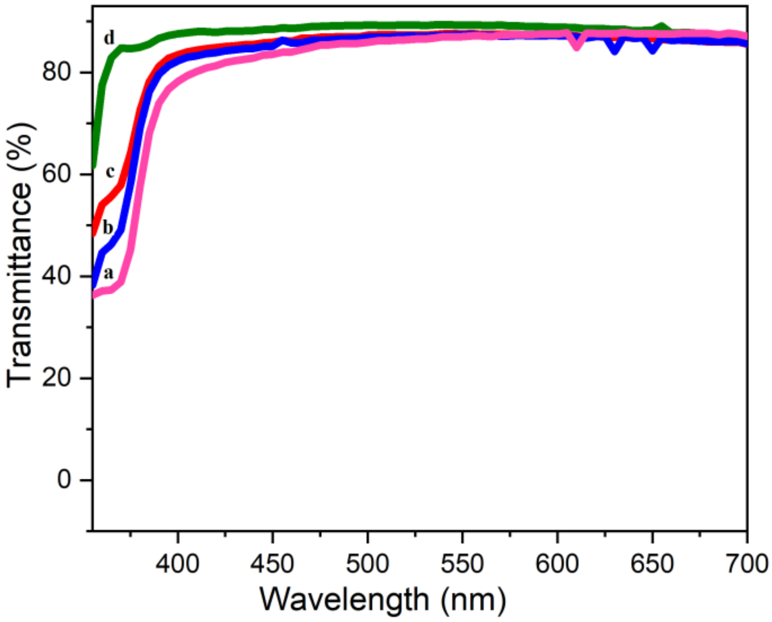

Figure. 9. *The transmittance spectra of P(NVC-co-BZMA) and it's composites; a) P(NVC-co-BZMA)/ZnO 15%, b) P(NVC-co-BZMA)/ZnO 10%, c) P(NVC-co-BZMA)/ZnO 5% and d) P(NVC-co-BZMA)*

When the absorbance-wavelength spectra of the P(NVC-co-BZMA) copolymer and composite samples were analysed, it was found that the absorption value of the P(NVC-co-BZMA) copolymer without any doping in the visible region was the lowest. The P(NVC-co-BZMA)/ZnO 15% composite with the highest doping ratio was found to have the highest absorption value. P(NVC-co-BZMA)/ZnO 5% and P(NVC-co-BZMA)/ZnO 10% composites were observed to exhibit absorption directly proportional to the doping amounts.

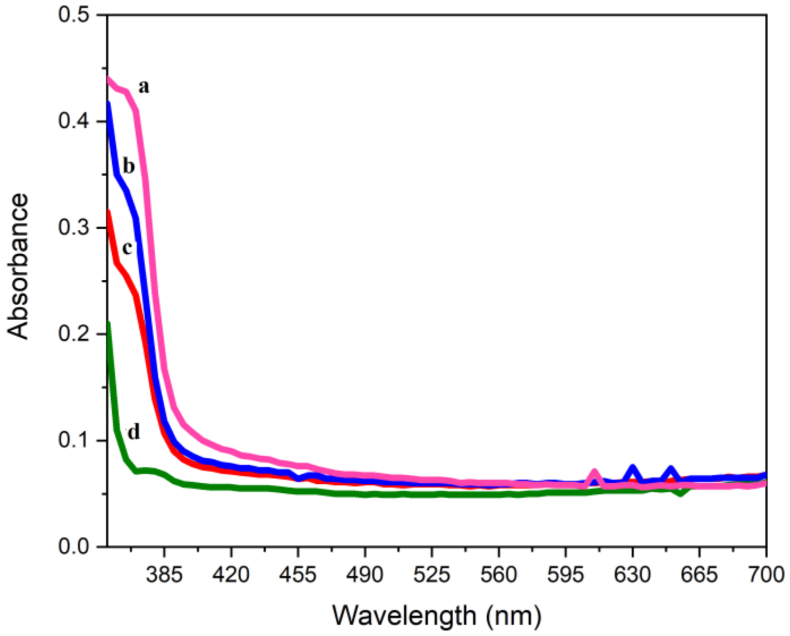

Figure. 10. *The absorption spectra of P(NVC-co-BZMA) and it's composites; a) P(NVC-co-BZMA)/ZnO 15%, b) P(NVC-co-BZMA)/ZnO 10%, c) P(NVC-co-BZMA)/ZnO 5% and d) P(NVC-co-BZMA)*

4. CONCLUSION

In this study, 25%-75% by mole copolymer of N-vinyl carbazole and benzyl methacrylate monomers and composites of this copolymer with nano ZnO in three different weight ratios were prepared. Thermal and optical properties of the synthesized samples were examined. The fact that the peak of the ester carbonyl was observed at 1723 cm^{-1} in the FT-IR spectrum of the copolymer and the absence of the peaks of the vinylic protons at 6.5 and 5.5 ppm in the ^1H NMR spectrum was important evidence that the copolymer was synthesized. All of the peaks belonging to the copolymer were also observed in the FT-IR spectrum of the composite. Thermal degradation behaviors and glass transition temperatures of the copolymer were determined by thermograms obtained from the TGA-DTA dual system. While the glass transition temperature for benzyl methacrylate polymer is 59°C in the literature, this value was determined as 227 °C for P(NVC) (Pearson & Stolka, 1981; Worzakowska, 2016). Glass transition temperatures of P(NVC-co-BZMA) copolymer were found in the range of those of P(NVC) and P(BZMA) homopolymers. The Tg value of P(NVC-co-BZMA) copolymer was 94.6 °C while the Tg values of the composites made with 5%, 10%, 15% ZnO doping were determined as 96, 98.4 and 99 °C, respectively. The Tg of the composites was higher than the Tg of the pure copolymer and it was determined that the softening temperatures increased as the nano ZnO ratio in the composites increased. This behavior shows that nano ZnO reduces the mobility of the chain in the copolymer chain, thus reducing the free volume of the polymer and increasing the softening temperature of the composites. While 310 °C defined the base temperature of the copolymer, the initial degradation temperatures of the composites decreased with the doping of nano zinc oxide into the polymer. In general, when the temperature values corresponding to 50% weight loss for the thermal stability of polymeric materials are evaluated, this value was determined as 382 ^{0}C for the copolymer, while it was 383.5, 385, and 427 °C for composites doped with 5%, 10% and 15% zinc oxide, respectively. Zinc oxide doped into the copolymer increased the thermal stability of the composites between 1.5 and 44 °C. The activation energy calculations of the P(NVC-co-BZMA) copolymer synthesized in the study were examined by employing the FWO and KAS methods. The activation energies for the copolymer were determined as 131.924 and 128.139 kJ/mol according to the FWO and KAS methods, respectively. The closest value to the average activation energy for the copolymer was obtained at a conversion value of 0.5. Activation energies of the P(NVC-co-BZMA)/ZnO 10% composite according to FWO and KAS method were calculated as 365.57 and 361.757 kJ/mol, respectively. The closest value to the average activation energy for this composite was obtained at a conversion value of 0.4. The UV spectra of the P(NVC-co-BZMA) copolymer and composites were analyzed in the visible region and it was observed that the undoped P(NVC-co-BZMA) copolymer had the highest optical transmittance and the lowest absorption value. As for the composites, it was determined that the optical transmittance values decreased with the increase in the amount of doped nano ZnO, while the optical absorption increased. In conclusion, it was observed that the absorption spectra were in agreement with the optical transmittance spectra.

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