

Thermogravimetric Analysis (TGA) and Determination of Activation Energies of Waste Paper Products

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Tetra Pak® paper and PE (polyethylene) wastes were evaluated to obtain a new composite material with awareness of environmental responsibility. Fillers and coupling agents were used for this joining process. Calcite (CaCO₃), boric acid (H₃BO₃), SPT (Sodium Perborate Tetrahydrate / NaBO₃·4H₂O), titanate, MAPE (maleic anhydride grafted polyethylene, and vinyltriethoxysilane (C₈H₁₈O) composites were produced in 30 × 30 cm plates. A total of 30 samples were prepared as 5 mg flour for thermogravimetric analysis (TGA). Samples were observed in a thermal analyzer at 10, 15, or 20 °C/min heating rate in nitrogen atmosphere. Graphs were obtained as a result of TGA, and the activation energies were calculated using these graphs. The activation energy was calculated using the Flynn-Wall-Ozawa method. The determined activation energies of the composites were close to each other for each temperature group. In the samples using calcite and vinyltriethoxysilane, relatively higher activation energies were observed in comparison to the samples with boric acid and MAPE.

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INTRODUCTION

Polymer materials made of paper, plastic, and aluminium are used widely as packaging materials, especially in beverage boxes. These materials are transformed into composite materials with the help of various additives and chemicals at the end of the usage process, and they are added to the industry as products with different usage areas.

Composites are heterogeneously structured materials consisting of more than two substances, which are insoluble in each other. At least one phase may be inorganic as a dispersed phase. Composites are designed to have superior mechanical properties and performance characteristics, regardless of their constituent materials. They can have unmatched advantages over single piece materials such as high resistance, high hardness, low density, long durability life, and high thermal stability, insulation, and transmission. They have a significant advantage in reducing life costs (Yılmaz 2008).

Whiskers used as fiber materials in composites are produced by crystallizing the materials in very small scales of dimension. Internal adaptation in each whisker is extremely high (Tiwari 2016). The matrix should be selected by taking into account the moisture and water absorption properties of the material. Properties such as viscosity, melting point, and temperature are important points for matrixes (Ulçay *et al.* 2002).

Matrix materials must have a high resistance to environmental factors such as water, chemicals, and UV in order to meet the requirements of the application areas of composites. Resins used in the production of fiber reinforced composite materials are sometimes referred to as polymers. Polymers are chain structures formed by simple molecular structures. Polymers are divided into two groups as thermoplastics and thermosets. Thermoplastics are structures that melt when heated and solidify when cooled. That is, they are readily recycled polymers. Nylon, polypropylene, polyethylene are examples of known thermoplastics (Ataş 2016). Waste Tetra Pak cartons are recycled by recycling the paper fibers in the paper to obtain different paper products. Waste Tetra Pak is divided into small pieces and pressed under temperature and pressure methods to produce chipboard (Megep 2017). Calcite is preferred as a filling material in plastics, with the advantage that the thickness remains homogeneous everywhere, and it has resistance to high temperatures, flexibility, and strength properties. In the paint industry, it is a preferred material for increasing the durability of the paint against water and chemical substances. In the paper industry, it is used to accelerate the drying of the ink by means of oil absorption. It is also used to prevent the spread of flame in other usage areas (Şahin 2017).

In this study, powdered calcite material was used to examine the positive properties of calcite in the produced composites. The plastic materials that are thrown into the environment are the wastes that need to be recycled because they remain for a long time without deterioration. The most consumed plastics are polyethylene (PE) and polypropylene (PP). With the recycling of waste plastic materials, advantages are provided such as energy saving, prevention of environmental pollution, and reduction of raw material requirements (Ay 2016). The waste plastics used in the study were thermoplastic based polyethylene. Due to hydrophilic properties for the hydroxyl groups in the cellulose fibers, when reinforced with the hydrophobic polymer, a poor interface between the fiber/matrix is formed and the moisture absorption resistance is also reduced. In natural fiber-containing composites, the binding agents have a main function of enhancing the interaction between functional groups of matrix material and cellulose OH groups (Bulut and Erdoğan 2011). The matrix material has the task of transferring the load to reinforcements, keeping them together and protecting them against environmental effects (Baş 2015). Polyethylene exhibits resistance to chemicals and processing temperature ranging from 150 to 250 °C, flame retardant, low water absorption, and it is a highly durable polymer with flexibility. Its typical properties include a specific gravity of 0.92 to 0.96 kg/cm³ and an elastic modulus of 1000 to 1500 MPa (Koluman 2018). It is an easy and inexpensive material to supply. In this study waste polyethylene is preferred. To connect polar wood materials and nonpolar thermoplastic polymers, matching chemicals and fillers were used at various ratios. This study is important because there are tons of waste paper waiting to be evaluated for utilization in our country. This work addresses the duration of use, sustainability, environmental sensitivity, and reduction of plastics and Tetra Pak wastes. Filling material, matrix and matching chemicals in the composite material produced in the study are shown in Fig. 1.

Thermal analysis methods date back a century. These methods include TG (Thermogravimetry) based on mass loss measurement, DTA (differential thermal analysis) and DSC (differential scanning calorimetry). The latter are two methods based on temperature difference. Changes in mass loss studied as a function of temperature or time are covered by the thermogravimetry method. With DTA, inorganic substances (ceramics, silicates, *etc.*) are examined, and it is aimed to determine the melting and boiling temperatures of organic substances. In addition, physical and chemical changes in

polymers are studied. Thermogravimetric analysis (TGA) involves a device that measures the change in mass of the material (Al-Ayed 2018). Thermogravimetry has an advantage over quantity at a constant temperature; some sample may change as the sample is heated to the desired temperature. Especially in the degradation of high polymers, the initial structure in the sample changes (Ozawa 1965). Activation energy was discovered by Hood about a hundred years ago as an experimental relationship dependent on temperature (Sudha *et al.* 2014). In this study, the calculated activation energy endpoint values were close to each other for each temperature group in the 10-15-20 °C samples. Activation energies were calculated with the help of the slopes of the graphs created with the second derivatives of the amount of change in the amount of substance with the effect of temperature with respect to time by thermogravimetric analysis.

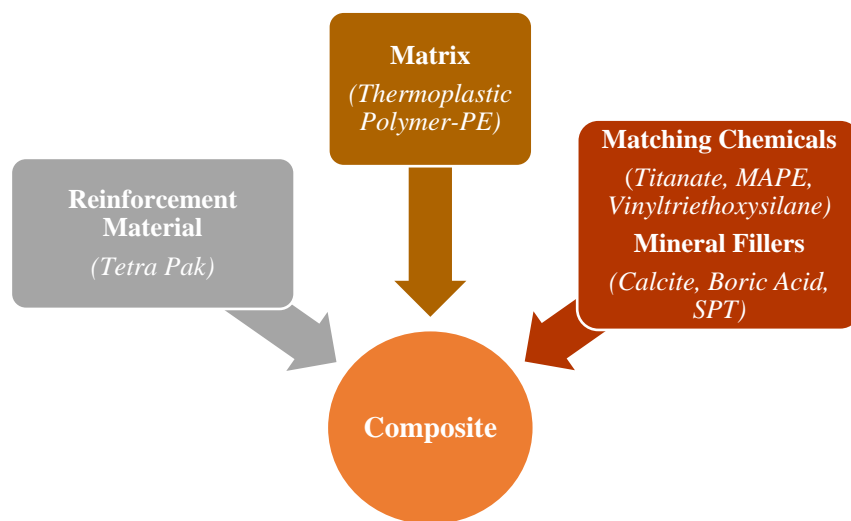


Fig. 1. Schematic representation of the composite material content produced

EXPERIMENTAL

Materials

Polymeric material-coated waste paper products (Tetra Pak®) were converted into small powder particles in a grinding machine with 1-mm blades. Prescription samples with different chemical substances were made from waste paper materials containing polyethylene, cellulose fiber, and a quantity of aluminium. The added substances were plastic material, calcite (CaCO_3), boric acid (H_3BO_3), sodium perborate tetrahydrate (SPT) ($\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$), titanate, acetic acid, maleic anhydride grafted polyethylene, and vinyltriethoxysilane. The coupling agents used were titanate, maleic anhydride grafted polyethylene (MAPE), and vinyltriethoxysilane ($\text{C}_8\text{H}_{18}\text{O}$).

A total of 30 composite materials in different groups of Tetra Pak®, Tetra Pak® + PE, or Tetra Pak® + PE + matching chemicals were produced in 1 x 30 x 30 cm plates in a press machine at 200 bar pressure, 170 ° C, for 10 min. In the first produced composite plate, 630 g of 10% (63 g) of plastic material was added to the milled Tetra Pak® material. To this material, 5%, 15%, 25% of matching chemicals were added to ensure bonding to the produced composite sheets.

Table 1. Production Conditions of Composite Sheets

Press Temperature/Lower Press-Upper Press (°C)	160-170
Press time (minutes)	10
Press pressure (bar)	200
Thickness (mm)	10
Sheet dimensions (cm)	1x30x30

Thermogravimetric Analysis (TGA) and Differential Thermogravimetry (DTG)

Thermogravimetry (TG) is based on mass loss, while differential thermal analysis (DTA) and differential scanning calorimetry (DSC) are based on temperature difference. The changes in mass loss observed as functions of temperature or time are in the thermogravimetric method (Ünlü 2008). The resulting mass losses are the dissociation of the substance depending on the temperature change or the separation of the volatile compounds such as usually water from the structure (Blaine and Hahn 1998).

For TGA, 30 types of composite materials were prepared as 5 mg flour. The thermal degradation and mass losses of the specimens were determined with an Exstar SII TG / DTA7200 model thermal analyzer at 10, 15, or 20 °C/min heating rate in a nitrogen atmosphere. The activation energies of the materials were calculated according to the following equation (Yakuphanoglu 2017).

$$E = - \text{slope} \times 8,315 \text{ (kJ/mol)} \quad (1)$$

where E is the activation energy and 8,315 is the gas constant.

The TGA was performed on composite materials prepared with different filling materials on 10, 15, and 20 grades of 30. Graphs were obtained, and the activation energies were calculated using these graphs. The results are shown in (Table 2). In terms of activation energies, approximately the same values were determined for each ratio using boric acid and MAPE. In the samples using calcite and vinyltriethoxysilane, relatively higher activation energies were observed than in the samples with Boric Acid and MAPE.

Thermogravimetric analyzes of 30 types of composite materials prepared with different fillers at 10, 15, and 20 degrees were made. Graphs were obtained, and the average values of activation energies (AE) were calculated using these graphs.

The kinetic behaviors of composite types gave similar results. The graphs below show the kinetic energies calculated with different temperature gradients in 30 samples in nitrogen atmosphere. The activation energy was calculated by the slope of the graph between the temperature in Kelvin in the x-axis and the logarithm of the temperature in the y-axis.

The interface between the reinforcing element and the matrix material affects the properties of the composite material. It increases the resistance against ignition by using powder calcite, which is used as reinforcing material in different proportions of the produced composite material types. Among the composite material types produced in the study, those produced with SPT have a disadvantage in that the mechanical strength is low. The desired binding properties with SPT have adversely affected the composite material in both physical and mechanical directions.

Table 2. Activation Energies of Composite Groups

EA	EA Means (J/mol.K)
1. Sample 25% Calcite 3% Vinyltriethoxysilane	28.360
2. Sample 15% Calcite 3% Vinyltriethoxysilane	28.349
3. Sample 5% Calcite 3% Vinyltriethoxysilane	28.339
4. Sample 5% Calcite 3% Titanate	28.348
5. Sample 15% Calcite 3% Titanate	28.322
6. Sample 25% Kalsit 3% Titanate	28.410
7. Sample 25% Boric Acid 3% Vinyltriethoxysilane	28.311
8. Sample 15% Boric Acid 3% Vinyltriethoxysilane	28.375
9. Sample 5% Boric Acid 3% Vinyltriethoxysilane	29.226
10. Sample 25% Boric Acid 3% Titanate	28.447
11. Sample 15% Boric Acid 3% Titanate	28.383
12. Sample 5% Boric Acid 3% Titanate	28.453
13. Sample 25% Calcite	28.502
14. Sample 15% Calcite	28.379
15. Sample 5% Calcite	27.924
16. Sample 3% Titanate	27.944
17. Sample Tetra Pak PE	28.028
18. Sample 1 Tetrapak	28.255
19. Sample 25% Boric Acid	28.033
20. Sample 15% Boric Acid	28.031
21. Sample 5% Boric Acid	27.974
22. Sample 25% Calcite 3% MAPE	27.969
23. Sample 15% Calcite 3% MAPE	28.001
24. Sample 5% Calcite 3% MAPE	28.041
25. Sample 25% SPT(No Acetic Acid) 3% Titanate	27.974
26. Sample 15% SPT(No Acetic Acid) 3% Titanate	27.998
27. Sample 5% SPT(No Acetic Acid) 3% Titanate	27.999
28. Sample 25% BA 3% MAPE	28.046
29. Sample 15% BA 3% MAPE	28.101
30. Sample 5% BA 3% MAPE	28.041

RESULTS AND DISCUSSION

Composite materials have a wide range of uses in furniture and construction. The properties of these products are improved by additives, which increase the resistance to water and fire. Moreover, additives change in the physical form of the raw material, plate density, and quantity of the glue used. The compatibilizer used in wood plastic composites plays an important role in bringing the compatibility to the maximum level between polar wood fibers and non-polar polymeric materials.

Thermogravimetric analysis (TGA) was used to measure the weight change of a material subjected to heat; in this study, 5 mg samples were heated in 5-degree increments. For each sample, temperature and mass loss graphs were obtained.

TGA is used to measure the weight change of a material subjected to temperature (Al-Ayed 2018). TGA tests were carried out with 5 mg samples and were heated in increments of 5 degrees and different temperature, and mass loss graphs were obtained for each sample. Activation energies were calculated with the help of the slopes of the graphs created by the second derivatives of the amount of change in the amount of substance with the effect of temperature with respect to time by TGA. The changes in temperature and substance content of sample (a) with Tetra Pak® + PE (a) sample, and sample (b) with Tetra Pak® + PE + 15% calcite + 3% MAPE by TGA are shown in Fig. 3.

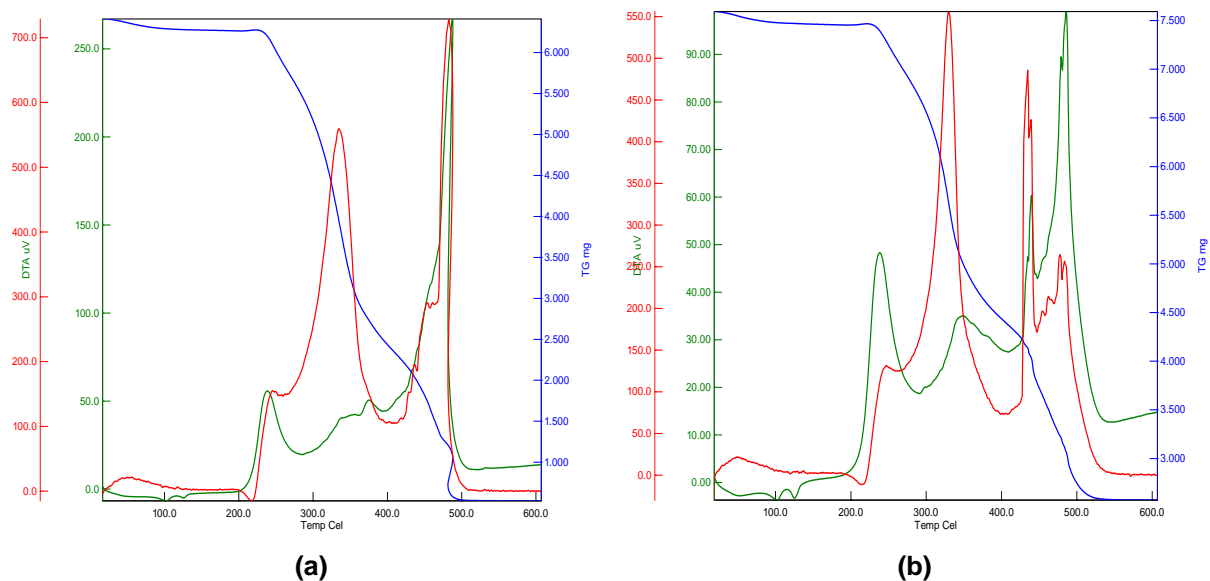


Fig. 2. (a) Change of temperature and substance content of Tetra Pak + PE sample with TGA (b) Change in temperature and substance content of Tetra Pak + PE + 15% Calcite + 3% MAPE sample with TGA

The change in temperature and substance content of Tetra Pak® + PE sample was analyzed based on the TGA findings. The change in the amount of substance (TG mg) in the Tetra Pak® + PE graph (blue colored curve) shows that approximately 6 mg of the sample was completely degraded before 500 °C. It was observed that the decrease in the amount of substance began at approximately 250 °C. It can be said that this temperature varied depending on whether a filler and matching chemical had been added to the sample. The DTG curve shown in red in the figure indicates that the rate of weight loss was a function of temperature and time (Düz 2018). The highest peak in DTG was observed around 480 °C. After this temperature value, a sharp loss of substance was observed. According to the change in temperature and substance content of Tetra Pak® + PE + 15% calcite + 3% MAPE sample with TGA, when the analysis result of the sample with MAPE and calcite was examined, it was observed that the amount of substance completely decomposed when it exceeded approximately 500 °C. The decrease in the amount of substance started at 200 °C. The DTG curve, on the other hand, showed different higher velocity values compared to the Tetra Pak® + PE sample without filler and matcher. This situation is thought to be caused by calcite and MAPE. The highest temperature value in DTG was seen around 330 °C. The second high velocity value was observed at 450 °C. The change in the amount of substance with temperature as a result of TGA of Tetra Pak®

+ PE + 15% calcite + 3% vinyltriethoxysilane and Tetra Pak® + PE + 3% titanate sample is shown in Fig. 3.

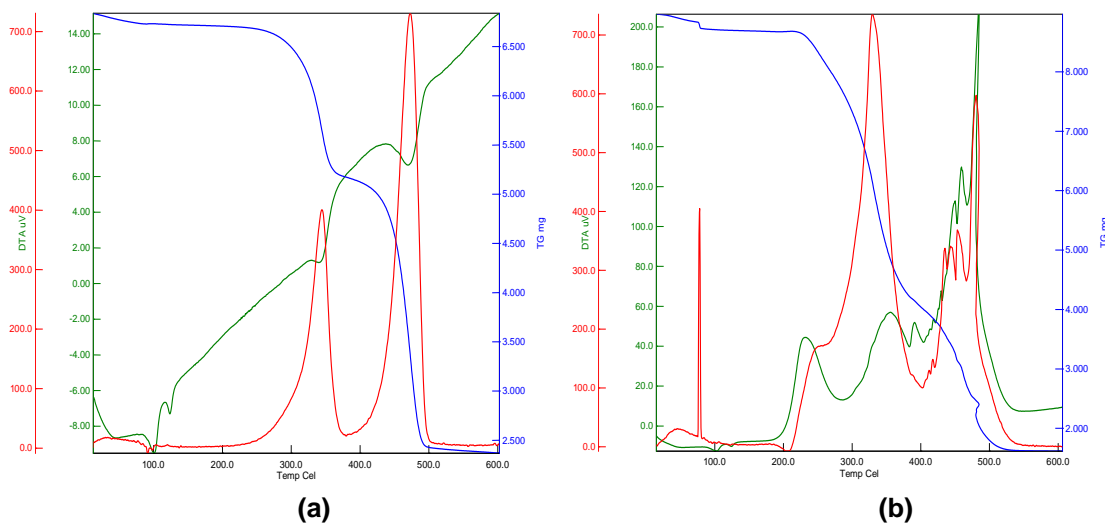


Fig. 3. (a) Change of temperature and substance content of Tetra Pak® + PE + 15% Calcite + 3% Vinyltriethoxysilane sample with TGA (b) Change of temperature and substance content of Tetra Pak® + PE + 3% Titanate sample with TGA

According to the temperature and substance content change of Tetra Pak® + PE + 15% calcite + 3% vinyltriethoxysilane sample with TGA, when the analysis result of the sample to which vinyltriethoxysilane and calcite were added was examined, it was observed that the amount of the substance was completely decomposed at temperatures close to 600 °C. It can be said that vinyltriethoxysilane coupling chemical increased the decomposition temperature value higher than the sample with MAPE. In this example, the DTG curve showed two mass loss steps at approximately 350 and 470 °C. The speed decrease observed at 100 °C indicates that one of the substances in its content was degraded and consumed. It is seen that the substance amount curve rose to higher temperatures when compared to other graphs. At the same time, more stable decreases were observed in the curves compared to other graphs. It can be said that the decomposition temperature increased when calcite and vinyltriethoxysilane were used together. As a coupling chemical, vinyltriethoxysilane can be said to provide resistance by showing good bonding. According to the change in temperature and substance content of Tetra Pak + PE + 3% titanate sample with TGA; In the sample to which titanate was added, the amount of substance began to deplete after exceeding 500 °C. According to DTG, the substance per minute (converted to grams) loss curve showed sharp velocity changes at different temperatures (90 °C to 350 °C). Compared to other graphs, a sudden mass loss was observed at 100 °C. This sample with titanate showed similar reaction properties to the sample using MAPE. The highest DTG peak was observed around 350 °C. Generally, it can be said that more resistance to degradation was provided in the sample using vinyltriethoxysilane. In another study, as a result of the thermogravimetric analysis, it was observed that the amount of Tetra Pak® substance ended at 500 °C. The cardboard layer deteriorated at a lower temperature than the plastic layer. Decomposition of polyethylene (PE) was shown at 475 °C maximum peak temperature, and the cardboard at 360 °C max peaked and showed a sharp weight decrease. It has been stated that the cardboard part of Tetra Pak® is mainly responsible for the carbonization condition. The mass loss seen

around 100 °C was caused by the reduction of water. It has been observed that aluminum has no effect on degradation. It has been stated that the degradation of cellulose is completed before the mass loss of polyethylene (Korkmaz *et al.* 2009). The graphs show that water was lost at about 100 °C. Unlike the others, the sample with titanate showed water loss with a sudden descent at around 100 °C. At this temperature, the rate of substance amount also showed a sharper peak compared to other graphs. In this example, it can be said that titanate and water did not show the desired harmony. According to another study, when the thermal behavior of wood and polymer mixtures was examined, it was observed that the mass loss percentage of cellulose reached the highest level at 326 °C, while the mass loss percentage of polyethylene reached the highest amount at 482 °C (Sharypov *et al.* 2001). The oven temperature in the device can be increased between 25 and 1600 °C for optional periods.

Crystallization in differential thermal analysis (DTA)

The DTA results showed exothermic, evaporation, and melting characteristics, which is expected for an endothermic physical event. Oxidation and polymerization are exothermic, while dehydration and degradation are endothermic. Nitrogen gas must be passed through the system in order to prevent combustion as a result of the sample in the device meeting with oxygen. As a result of the analysis, the data is transferred to a graph with the software. Temperature is shown on the X axis and the change curves of other temperature-related parameters can be revealed by the software. In order to calculate the activation energies, there must be at least three analysis curves at different temperatures (Ünlü 2008).

The kinetic behaviors of different composite types were similar. In the graphs to be shown, the kinetic energies calculated with different temperature gradients in nitrogen atmosphere are shown. In the graphs, the activation energy is calculated by the slope of the graph between the temperature in Kelvin in the x-axis and the logarithm of the temperature in the y-axis. 1,890,000 is the independent coefficient in Arrhenius temperature integral (Tang *et al.* 2003). Figure 4 shows a kinetic analysis curve in the nitrogen atmosphere of 5% calcite + 3% vinyltriethoxysilane composite sample.

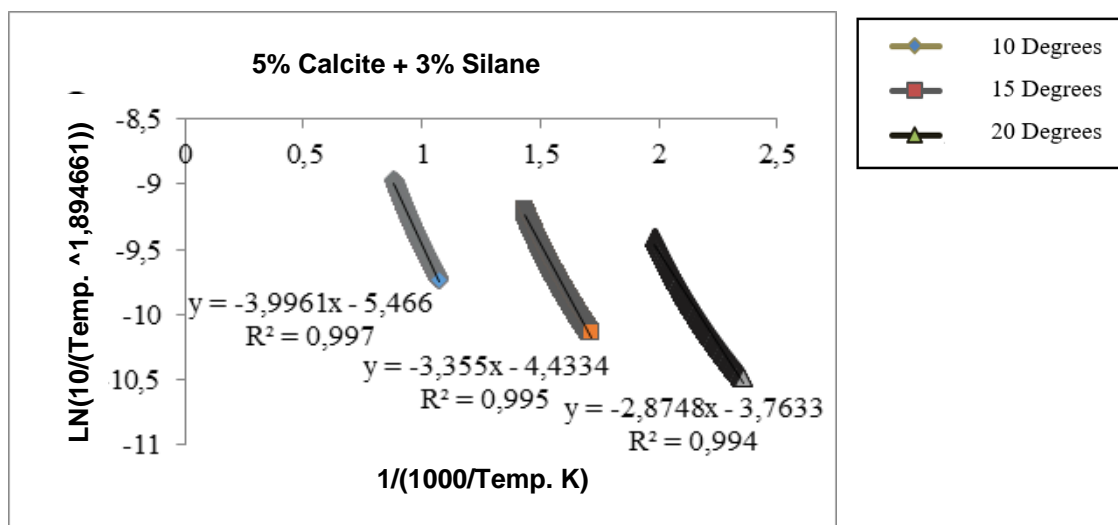


Fig. 4. Kinetic analysis curve of composite sample 5% calcite + 3% vinyltriethoxysilane in nitrogen atmosphere

The activation energies were calculated from the slopes of the graphs generated by the second derivative of the amount of change in the amount of the substance. At the same time, most samples were degraded and depleted at 600 °C. In the sample containing only Tetra Pak® was decomposed at 100 °C, 15% boric acid 3% titanate at 300 °C, 15% boric acid 3% vinyltriethoxysilane at 400 °C, 5% calcite, 3% titanate, Tetra Pak® and PE, 25% calcite 3% MAPE, 15% calcite 3% MAPE, 5% calcite 3% MAPE at 550 °C, and 5% calcite 3% MAPE, 15% calcite 3% MAPE, 25% calcite 3% MAPE samples at 900 °C.

TGA of paper cup waste was determined at 25 °C/min, 30 °C/min in air, and 30 °C/min in nitrogen atmosphere. Activation energy was detected using thermogravimetric curves. As a result, the activation energy increases from 17 to 28 KJ/mol with increasing heating rate from 25 to 30 °C/min in the air atmosphere. Activation energy has been reported to be less (22 KJ/mol) in a nitrogen atmosphere in comparison to 28 KJ/mol in air atmosphere at a heating percentage of 30 °C/min (Singh *et al.* 2012).

In another similar study, when the TGA results of the boards produced from chips modified with vinyltrimethoxysilane were calculated, it was observed that the degradation started at 200 °C (Gönültaş *et al.* 2022). However, in this study, it can be said that when calcite and vinyltriethoxysilane are used together, the decomposition temperature increases (350 °C to 470 °C). According to a study on the thermal and combustion properties of wood-plastic composites, it was understood that as the amount of boron compounds increased, the burning degrees (°C) of the lignocellulosics and plastic materials in the composite material increased. In addition, it was understood that the horizontal burning rate decreased as the amount of boron added to the composites increased (Altuntaş *et al.* 2017). Likewise, in this study, we can say that vinyltriethoxysilane added to the composite material slowed down the combustion.

CONCLUSIONS

1. Additives and coupling agents were found to affect the physical and mechanical properties of the composites. The product performance was improved by modifying these additives or by combining chemistries.
2. Thermogravimetric analysis showed that the fillers had no significant effect on these temperature values. In terms of thermogravimetric analysis (TGA), it was determined that the activation energies of the experimental groups gave close values in different temperature groups.
3. Complete degradation was observed due to the addition of MAPE and calcite in the change of temperature and substance content.
4. As a result of TGA, it can be said that the decomposition temperature increased when calcite and vinyltriethoxysilane were used together. As a coupling chemical, vinyltriethoxysilane can be said to provide resistance by showing good bonding.

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