Effect of Melt Temperature and Holding Time on Mechanical Properties of Polypropylene Composites Bottom Ash Reinforced

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Abstract. In this study, observing the effect of variations in melt temperature and holding time on the mechanical properties of polypropylene composite materials with coal bottom ash reinforcement has been done. This composite used a particle size of 200-250 mesh with a composition of 10%, and a polypropylene matrix of 90% by weight. Melting temperatures had a variation of 170°C, 180°C and 190°C, and holding times had a change of 0, 30 and 60 minutes. Bottom ash was washed with fresh water (room temperature) and warm water (80°C) until clean from impurities. The next process, bottom ash was drained at room temperature for 24 hours and dried at a temperature of 120°C for 3 hours. The test results showed the value of composite increases to 24.4% for tensile and 123% for flexure strength from the pure matrix strength. These properties were essential information for alternative engineering material research.
Keywords: glucose-fructose syrup, fructose, ion chromatography.

1. Introduction
The rapid development of technology and manufacturing requires a lot of materials of various types, both metal, wood and plastic. However, the metal and wood materials begin to be abandoned because of supplies in nature decrease [1]. Currently, plastic is widely used as an alternative material for engineering materials [2]. At this time also begin to develop public awareness using materials that are environmentally friendly, recyclable, inexpensive, easy to process, and have a lot of supplies, [1,3-7].

Many studies in the field of composites have been carried out related to methods, processes, parameters and types of matrix and reinforcing materials. Especially for the matrix and reinforcement materials can be sourced from metal, synthetic, plastic, and natural materials. Polypropylene is often used as a matrix because it is widely used in daily life, has good mechanical properties (strength and stiffness), sufficient process temperature and can be recycled [7]. As for reinforcing materials, many researchers use materials from a waste of coal combustion. The combustion process will produce quite a lot of waste, which can pollute the environment. The wastes are in the form of fly ash and bottom ash which they are often studied as reinforcing polymer composites, as has been done by [8,9,10,11,12].

Researches on polymer composites with particle and fibre reinforcement have been widely reviewed from various parameters. [6,12, 13] stated that the size, surface conditions of the particles are very influential on the attractiveness of the interface that affects the performance of the composite. Whereas [10], found that the bottom ash content had more influence on the mechanical properties of polymer composites. In research conducted by [2] found that the melting temperature of the polypropylene matrix and the duration of stirring affect the mechanical properties of composites. [14] also stated that
the increase in temperature in the process causes a decrease in the strength of polymer matrix composites. [15] indicated that melt temperature affects the final molecular weight of the product, which has an impact on the mechanical properties of the composite. In [16] conveyed that the time and temperature in the process of the making polymer composite affect the mechanical properties. The same thing was expressed by [17], that the increase in melting temperature allows the process of solidification to take longer and encourage the growth of gas cells in the matrix, which can trigger the appearance of pores on the composite. This condition will affect the strength of the composite. Under these conditions, this study will observe the effect of variations in melting temperature and holding time in the process of the making composite on mechanical properties. These results will be the basis for developing composite materials, so as to obtain composite materials that are suitable for technical requirements.

2. Experiment

2.1. Polypropylene Matrix
Polypropylene (homopolymer) was obtained from PT. Politama Propindo, Surabaya-Indonesia in the form of granules on 25 kg packaging. The mechanical properties of polypropylene at room temperature are tensile strength of 34 MPa, modulus of elasticity of 1325 MPa, flexure strength of 41 MPa and modulus of flexure of 1550 MPa.

2.2. Bottom Ash Reinforcement
Bottom ash was given by CV. Partika Arnawa, Sidoarjo-East Java, in the form of small chunks and still mixed with impurities. Bottom ash was dried by the sun. Then, the bottom ash was crushed into particles and sieved to get a size of 200-250 mesh. The Particles had been washed in freshwater (room temperature) until clean, before they were washed with warm water (80°C) for 3 hours to remove the oily parts, while stirring and the floating parts were removed. The next process, the particles had been drained in an open room (room temperature) for 24 hours, before they were dried in a heating container with a temperature of 120°C for 3 hours, while stirring slowly to dry completely. Finally, the particles were ready used as reinforcement.

2.3. Making of Composite
The compositions of the composite are polypropylene of 90%, and bottom ash of 10% by weight as same as the weight percentages of particles are used by [18,19]. The matrix had been melted 100%, the bottom ash was introduced and stirred at a speed of 20 rpm for 30 minutes. The variations of melting temperatures were 170°C, 180°C, and 190°C. A mixture of the composite was printed on wood mould that it had been provided. The mix of composite material had been given a holding time, before it was poured into a mould. The variations of holding time were 0, 30, and 60 minutes. Each holding time had range of 30 minutes, and the composite mixture was obtained a stirring interrupted for 5 minutes with a speed of 30 rpm. Furthermore, the composite material in the mould was given the pressure of 25 kg/cm² for 5 minutes. Finally, the material was removed from the pattern and processed into test specimens.

2.4. Mechanical Testing
Test specimens were as per ASTM D638 for tensile test, and as per ASTM D790 for 3 points bending test. The results of the test formed load and extension relationship diagrams, which were then processed to get the tensile and flexure strength, strain, and modulus values.

3. Results and Discussion

3.1. Tensile Strength
Figure 1 explain the effects of variations on melting temperature and holding time on the tensile strength of composites. It is seen, the higher of melting temperature has an impact on the decrease in tensile strength. Likewise, the holding time has the same effect, namely reducing the strength of the composite. This decrease in strength is more influenced by changes in polypropylene conditions, which are increasingly brittle, and there are many pores in the composite cross-section. With a higher melting temperature, polypropylene more easily to form gas in the solution, so the bubbles are trapped in them and becomes pore while cooling. The same condition occurs when giving holding time is getting longer.

![Graph](image)

**Figure 1.** a). Effect of melting temperature on tensile strength, b). Effect of holding time on tensile strength.

The best tensile strength is obtained at a melting temperature of 170°C with a holding time of 0 minutes. Fig. 1a and 1b show that the best tensile strength for all variations in melting temperature is obtained at 0 minutes holding time. Likewise, for all variations of holding time, the best value is received at a melting temperature of 170°C. All of these values are above the tensile strength value of pure polypropylene.

Based on observations, the stirring process also triggers bubbles in the mixture. The stirring process causes air to be trapped in a thick polypropylene solution, and it is difficult to escape so that it becomes pores when cooling. From this study, the lowest tensile strength value are obtained at a melting temperature of 190°C with a holding time of 60 minutes. This tensile strength decreases about 33.6% of the strength of pure polypropylene. Fig. 1a also shows that the tensile strength at melting temperatures of 180°C and 190°C does not significantly affect on holding time of 30 and 60 minutes. The results show that the tensile strength of the composite is more impact on holding time than the melting temperature.

### 3.2. Modulus of Elasticity

Figure 2 shows that the modulus of elasticity is affected by changes in melting temperature and holding time significantly. In general, the modulus of elasticity of composite decreases when the melting temperature and holding time increase. The modulus of elasticity indicates the level of material ability to deform elastic or the level of stiffness. This decreasing in value indicates that the composite is more ductile in line with increasing a melting temperature and a holding time. In this condition, the reduction in the modulus of elasticity is more due to a decrease in the ability to accept tensile strength rather than an increasing elastic strain, see in Fig. 3.

Provision of holding time aims to provide an opportunity for the matrix to infiltrate more in-depth into the cavity or pore of the particle, it is hoped that the bonding of the interface will be better.
Likewise, with an increase in melting temperature, it is expected that the polypropylene matrix melts with a lower viscosity value, that it is easier to enter the cavity or pore of the particle. However, the matrix condition does not have an impact on the interface bond and the ability of composite deformation because the degradation of the matrix is more dominant. Interesting things are shown in Fig. 3a, that elastic strain does not show a regular pattern due to changes in melting temperature or holding time. This condition could be due to the impact of the appearance of pores or the matrix ability decreasing. The number of pores in the cross-section area is varied and unpredictable, so the composite properties are not similar. Fig. 3b shows a more regular trend of yield strength, which has a decreased tendency due to the addition of melting temperature.

3.3. Flexure Strength

Figure 4 shows the flexural strength value of the composite material due to changes in melting temperature and holding time. It can be seen that the holding time is more influential than the melting temperature. So, the holding time has a significant impact on flexure strength. The holding time affects the matrix and bottom ash conditions. And in this study, there is the interrupting stirring which also triggers the emergence of pores and it impacts on the composite strength.

Figure 4a shows that the greatest flexural strength occurs at a holding time of 0 minutes for all melting temperature variations. This value also has a significant difference compared to the holding time of 30 and 60 minutes.
Figure 4. Flexure strength values due to the effect of melting temperature and holding time

Whereas, Fig. 4b shows that the flexural strength at a melting temperature of 190°C is highest under all conditions holding time. This condition is caused by the temperature to decrease the matrix's ability, especially in the property of plasticity, which causes rigidity of the composite increasingly. This situation will be very influential during the bending process, which has two sides, these are pressure and tensile side. Where it is compressive side becomes better so that the load on the bending process is higher. The low deformation on the compressive side has a good effect on the tensile side because the strain on the tensile side is increasingly difficult. The flexural strength increases to 91.6 MPa compared with pure polypropylene which is 41 MPa, or it increases about 123%. This value occurred at a melting temperature of 190°C with a holding time of 0 minutes.

3.4. Modulus of Flexure

Figure 5 shows how the temperature and holding time affect the flexure modulus value. Higher melting temperature causes greater material stiffness, see Fig. 5a. This condition occurs because the matrix has decreased deformation, which creates a higher rigidity of the material. This property will provide an increased ability to fight the bending. Whereas Fig. 5b shows the effect of holding time on the flexure modulus value, wherewith increasing holding time makes the composite stiffness smaller. The reduced modulus of flexure due to the ability of the composite to accept bending loads decreases. This condition, due to the presence of quite a lot of pores in the cross-section and particle has degradation. So, the composite elasticity does not significantly increase, but the ability of the composite to accept a load is reduced.
3.5. Morphology Analysis

**Pores.** Figure 6 shows the pores in the cross-section of the fracture area, which is in line with the increase in melting temperature. This fact is consistent with [17] that melting temperature triggers the emergence of gas cells in the solution that becomes bubbles during the composite manufacturing process.

![Figure 6](image)

**Figure 6.** The presence of pores in the cross-section of the tensile test results. Composite conditions at melting temperature variations with the same holding time (30 minutes).

Bubbles will turn into pores on the cross-section when the composite is cold. The impact of the existence of these pores is reducing the active area that it affects the composite strength. On the other hand, the holding time that is given at the time of process also has the effect of increasing the pores on the composite cross-section, see Fig. 7. The presence of interrupted stirring at a specific range contributes to the increase of bubbles in the composite solution. From this condition, the higher of melting temperature and holding time will trigger the appearance of more pores on the composite cross-section. This state impacts on tensile and flexure strength, as discussed above.
0 minutes holding time  30 minutes holding time  60 minutes holding time

Figure 7. Increasing pores condition in the cross-section of tensile test results for melting temperature of 180°C with the difference of holding time.

Bottom Ash condition. Figure 8 shows the condition of the bottom ash being melted at 170°C with a holding time variation.

![Figure 8](image1.png)

(a) (b) (c) (d)

Figure 8. The surface condition of the cross-section of test specimen at Tm 170°C. a). Distribution of particles at holding time of 0 minute, b). State of bottom ash particle at holding time of 0 minutes, c). Distribution of particles at holding time of 60 minutes, d). Condition of bottom ash particle at holding time of 60 minutes.

The state of particles with a holding time of 0 minutes, shows that the particles are more intact, see Fig. 8a and 8b. It is compared to particle conditions given a holding time of 60 minutes, see Fig. 8c and 8d. The particles are more fragile and become small pieces, which can make the composite strength decreases. The distribution of particles occurred fairly evenly at both the holding time of 0 and 60 minutes, see Fig. 8a and 8c. These results indicate that the stirring process can distribute the particles by well.

Figure 9 shows the condition of the particles in a composite cross-section with a melting temperature of 190°C and holding time of 0 and 60 minutes. The results at high melting temperature show many pores on the surface of a composite cross-section. While, the impact of the holding time of 60 minutes shows the state of particles that are not intact, Fig. 9d.
Figure 9. The surface condition of the cross-section of a test specimen at Tm 190°C. a). The state of particle distribution at 0 minutes holding time, b). The condition of Bottom ash particle is more intact at 0 minutes holding time, c). The state of particle distribution at 60 minutes holding time, d). The condition of particle breaks at 60 minutes holding time.

Based on these observations, to get a composite with good mechanical properties, following the wishes, it is necessary to consider several things, namely the melting temperature level, the length of holding time and the method of mixing composite materials.

4. Conclusion

In this study, it is showed that variations in melting temperature and holding time have a significant impact on the mechanical properties of polypropylene composites with bottom ash reinforced. The holding time has a more dominant effect on the mechanical strength of the composite compared to the melting temperature.

The higher degree of melting temperature will reduce the tensile strength, but increase the flexure strength, whereas the length of the holding time causes the tensile strength and flexure to decrease. On the other hand, the higher melting temperature drops the modulus of elasticity but tends to increase the modulus of flexure. Meanwhile, the longer holding time will reduce the value of elastic modulus and flexure modulus.

Physically, the quality of composite is influenced by increased melting temperature and holding time, which triggers to a growing number of pores and affects the integrity of bottom ash particles. So, a degree of melting temperature, the length of holding time and the method of mixing the composite material are considered for acquired an optimal result. This result will be used as a reference in the next research to find new material.

5. References
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