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THE IMPACT OF PROCESSING CONDITIONS ON THE MECHANICAL PROPERTIES, HARDNESS, AND STRUCTURE MORPHOLOGY OF POLYPROPYLENE/SILICA SAND POWDER

NGHIÊN CỨU KHẢO SÁT ẢNH HƯỞNG ĐIỀU KIỆN CHẾ TẠO VẬT LIỆU COMPOMZIT POLY PROPILEN/BỘT CÁT SILICA VÀ TÍNH CHẤT CỦA CHÚNG

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ABSTRACT

The polypropylene/silica sand powder polymer composite materials were fabricated by using the melt mixing method in a Haake internal mixer in the range of 170°C - 200°C. The polypropylene/silica sand powder polymer composite materials were fabricated by using the melt mixing method in a Haake internal mixer with varied conditions by adjusting the temperature range from 170°C to 200°C, rotor speed from 40 - 70rpm, and fusion time of 3 - 5 min. Torque visco of materials was also recorded by Haake Polylab software during melt mixing. The result indicated that uniformly dispersed materials at 50rpm for 5 min produced good mechanical properties with a significant improvement in hardness. Fourier transform infrared spectroscopy, mechanical properties, and scanning electron microscopy (SEM) of the materials were investigated.

Keywords: *Poly propyle (PP), silica sand powder, Shore D, PP/BCSi polymer composite.*

TÓM TẮT

Đã nghiên cứu khảo sát ảnh hưởng của nhiệt độ trộn, tốc độ trộn và thời gian trộn đến tính chất lưu biến, tính chất cơ học và hình thái cấu trúc của vật liệu compozit PP/BCSi. Kết quả thu được khi tăng nhiệt độ từ 170°C đến 200°C tính chất lưu biến nóng chảy của vật liệu giảm nên khả năng gia công dễ dàng hơn. Tuy nhiên, khi ở 200°C tính chất cơ học của vật liệu giảm, ở 170°C, 180°C khả năng phân tán thấp, lưu biến nóng chảy của vật liệu cao khó gia công so với ở nhiệt độ 190°C. Khi tăng tốc độ trộn kết quả cho thấy với tốc độ trộn chậm hoặc tốc độ trộn nhanh khi chế tạo vật liệu đều làm cho khả năng phân tán, tính chất cơ học của vật liệu compozit thu được thấp hơn ở tốc độ trộn 50 vòng/phút. Khảo sát ảnh hưởng của thời gian trộn thu được với thời gian trộn 5 phút hệ phân tán đồng đều, vật liệu thu được có tính chất cơ học tốt. Vật liệu compozit Poly propylen với bột cát silica thương mại của Việt Nam (PP/BCSi = 70/30 %kl) được chế tạo bằng phương pháp trộn nóng chảy ở 190°C với tốc độ trộn 50 vòng/phút trong thời gian 5 phút trên thiết bị Haake Rheomixer dễ gia công và có tính chất cơ học tốt, đặc biệt độ cứng của vật liệu tăng lên đáng kể.

Từ khóa: *Poly propyle (PP), bột cát silica, độ cứng Shore D, compozit PP/BCSi.*

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1. INTRODUCTION

Polypropylene (PP) has become one of the grow rapid plastics because of its balanced physical and mechanical properties. It can be heated, cooled, and melt without causing a significant reduction in its base composition. Being thermoplastic in nature, PP is considered common use in many industries because of its physical properties, adaptability, and environmentally friendly record due to its recyclability. Moreover, it has good thermal stability, chemical resistance, mechanical strength, and low cost [1-3]. Nevertheless, low-impact strength and toughness, especially at low temperatures, limited some of their industrial applications. In order to improve PP toughness, it has been studied and modified hard inorganic fillers [4], combined with thermoplastics [5], forming in situ microfibre or liquid crystal reinforcement [6] and nucleation for PP. The method of adding inorganic fillers is an effective way to enhance some of the mechanical properties of PP [4].

Silica has been widely used as reinforcement in polymers for several reasons: its excellent bioactivity and provides stability to the particle in changing environmental conditions. Several studies indicated that by adding nano-silica to the thermoplastic, the mechanical properties such as hardness, mechanical strength, elastic modulus, hysteresis, and fire resistance,... are significantly improved [7]. Some studies used fly ash combined with PE by the hot fusion method, which has improved from the observed level and chemical stability of PE/fly ash composites [8]. Furthermore, after the modification of silica by organic silane, the silanol groups on the surface allow for easy functionalization of the particle by covalent bonding of other ligands through different chemical reaction pathways or to form hydrogen bonds between inorganic and organic. As a result, the properties of materials were enhanced.

The production of sand powder is simple because it includes, at most, only washing and classification into

grades differing in grain size, so silica sand powder is an abundant, easy-to-find material in Vietnam with high silica content. Silica sand powder is a raw material applied in fields such as glass production, foundry, chemical production, paint and coating industry, raw materials in the ceramic industry, refractory materials, additives and fillers in construction, paper and plastic industries, oil and gas recovery and other applications [9, 10]. However, studies use silica sand powder as reinforcement for thermoplastics, thermosetting plastics, and rubber are not many. Therefore, in this study, we present the results of investigating the influence of processing conditions for instance temperature, time, and mixing speed rotor on the mechanical and physical properties of composite materials based on thermoplastic PP and sand powder as well as their structural morphology.

2. EXPERIMENTAL

2.1. Materials

Polypropylene (PP) resin was used in the form of commercial product granules (Singapore) with a density of 0.903g/cm^3, a flow index of 20g/min at 146°C.

Silica sand powder (BCSi) commercial product (Vietnam) with SiO_2 content 99.0 - 99.8%, $Fe_2O_3 \leq 0.03\%$ with size 5µm.

Other chemicals.

2.2. Sample preparation PP/silica sand powder composite

PP/BCSi samples were prepared using the melt mixing method in a Haake internal mixer. The mixture of PP and BCSi was mixed in a ratio of 70/30 and accurately weighed to ensure the fill factor of the sample in the mixing chamber was 0.75. The manufacturing conditions are shown in Figure 1.

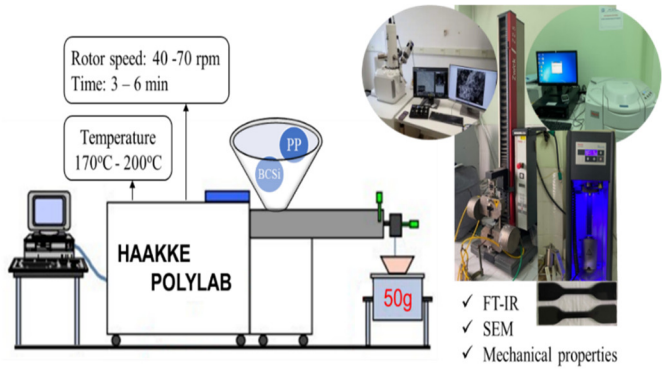

Figure 1. Fabrication diagram of PP/BCSi composite materials

2.3. Characterization

2.3.1. Fourier Transform Infrared Spectroscopy

The FTIR spectra of the samples were recorded using Fourier Nexus 670 (USA) at room temperature, in the wavenumber region from 500 to 4000cm^{-1} with a 4cm^{-1} resolution and 32 scans by using the reflexed method.

2.3.2. Scanning Electron Microscopy (SEM)

Morphology of the samples were analyzed by using a field emission scanning electron microscopy (JEOL, JSM-6510LV, Japan) at Institute of Materials Science, Vietnam academy of science and technology. The samples were coated with platinum prior to SEM observation.

2.3.3. Melting rheology

Melting rheology data was collected using Polylab simulating software connected to Hakke polylab os Determine the temperature variation and the force of the rotor following the time, thereby determining the melting rheological state of the material.

2.3.4. Mechanical properties

The tensile properties and elongation at break of the testing samples were performed on a universal testing machine (Zwick V.2.5, Germany) at the room temperature with a crosshead speed of 50 mm/min, in standard with ASTM D638M-93/89 for plastic

2.3.5. Determine firmness properties

The firmness of the material was measured on Durometer device (Germany), shore D according to ASTM D785-08 standard.

2.3.6. Determination of impact resistance

The impact strength of the material was conducted according to ISO 179 standard on the impact measuring device of Olsen Tinus (USA).

3. RESULTS AND DISCUSSION

3.1. Investigate the effect of mixing temperature

The investigation of the influence of temperature on the fabrication of PP/BCSi composite materials was carried out using a Haake internal mixer from the USA. The mixer was set to a time of 3 minutes and a mixing speed of 50rpm. The materials were tested at temperatures of 170°C, 180°C, 190°C, and 200°C. The viscoelasticity of polymers was analyzed by observing the instantaneous torque variation of the material with time in the molten state. Figure 2 displays the torque diagram of the PP/BCSi composite materials fabricated at different temperatures.

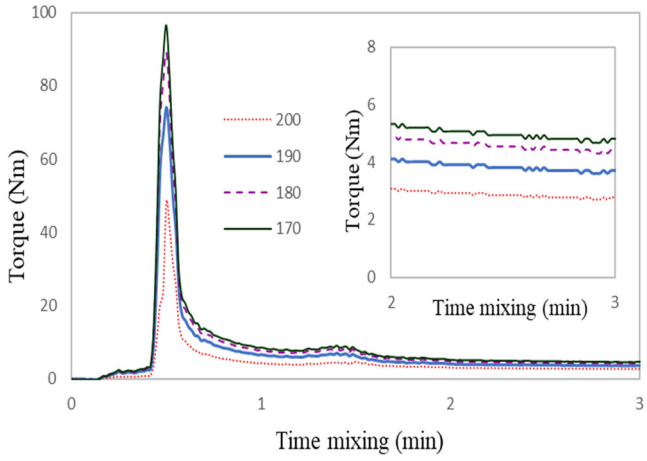

Figure 2. The torque behavior of PP/BCSi was observed during several different time intervals of mixing

Based on the result above diagram shows that the torque of the samples was highest during the initial phase when the

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mixing chamber was closed and the solid-state materials were being processed. However, as the PP melt began, the torque gradually decreased and eventually reached a steady state after three minutes of mixing time. This steady state corresponded to a constant torque.

During the molten state, the BCSi particles were able to disperse into the middle of the PP chain, which reduced the mobility and flow of PP. As the temperature increased, the torque of the PP/BCSi materials decreased, making it easier to process. On the other hand, when considering the mechanical and physical properties of the formed materials (as shown in Table 1) that indicated the mixing temperature of 190oC was the most suitable condition for fabricated PP/BCSi composite materials.

Overall, these findings suggest that the torque of the materials during processing can be used as an indicator of their suitability for specific applications. By carefully controlling the mixing temperature, optimizing the properties of the composite materials, and ensuring that they meet our desired specifications.

Table 1 presents the mechanical and physical properties of PP/BCSi composites, which were fabricated at various temperatures.

Table 1. The impact of temperature during mixing on the mechanical properties of PP/BCSi composite materials

Temperature (°C)	170	180	190	200
Tensile strength (MPa)	18.34 ± 1.1	22.16 ± 1.2	24.00 ± 1.0	20.32 ± 0.9
Elongation at break (%)	389.61 ± 2.7	401.87 ± 3.0	414.57 ± 2.3	387.54 ± 2.5
Impact strength (kJ/m^2)	20.78	24.56	27.83	22.78
Shore hardness (Shore D)	66.02 ± 2.0	70.43 ± 2.2	74.59 ± 2.4	67.54 ± 2.0

According to Table 1, we can observe that the tensile strength and flexural strength of the materials are at their highest level when the fabrication temperature reaches 190°C. This is due to the better melting capacity of the PP, which enhances the dispersion process and results in a more uniform distribution. However, it's important to note that if the temperature were to increase beyond 190°C to 200°C, the mechanical properties of the composites would deteriorate. This is because the PP macromolecule chain would decompose, resulting in a reduction in the product's mechanical strength.

3.2. Investigate the effect of mixing time

Figure 3 illustrates the impact of the mixing time of PP with 30% BCSi at 190°C on the tensile strength and elongation at break of the PP/BCSi composite.

Figure 3 shows that the highest values for tensile strength and elongation at break are achieved with a mixing time of 5 minutes. If the mixing time is less than 4 minutes, there may not be sufficient dispersion and even distribution of BCSi into the PP. Conversely, if the mixing time exceeds 5 minutes, it could lead to thermal oxidation decomposition reactions and the breaking of macromolecule chains that lead to reduced material mechanical properties. Therefore, it is crucial to maintain an optimal mixing time to achieve the desired mechanical properties of the material.

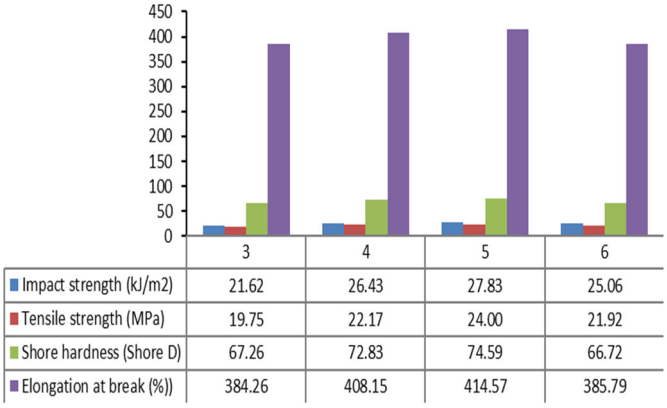


Figure 3. The impact of mixing time on the mechanical durability of PP/BCSi composite samples

3.3. Investigate the effect of rotor speed

Table 2 shows the impact of mixing speed in conjunction with 30% BCSi at a temperature of 190°C for 4 minutes on the tensile strength and elongation at the break of the PP/BCSi composite.

Table 2. The impact of rotor speed on the mechanical properties of PP/BCSi composite samples

Mixing Speed (rpm)	40	50	60	70
Tensile strength (MPa)	21.09 ± 1.1	24.00 ± 1.0	23.47 ± 1.2	23.05 ± 1.1
Elongation at break (%)	392.71 ± 2.6	414.57 ± 2.3	408.56 ± 2.4	402.21 ± 2.7
Impact strength (kJ/m^2)	23.19	27.83	26.35	25.96
Shore hardness (Shore D)	68.18 ± 1.9	74.59 ± 2.4	71.34 ± 2.2	70.32 ± 2.0

According to the data in Table 2, the PP/BCSi composite materials exhibit the highest tensile strength and elongation at break when mixed at 50rpm. At mixing speeds below 50rpm, the dispersion and distribution of BCSi in PP are not uniform, and the interaction between the two materials is low, resulting in an inconsistent material structure. However, higher mixing speeds above 50rpm increase torsion, compression, and shear forces, causing a reduction in material viscosity, breakage of PP macromolecule chains, and clustering of BCSi in the material.

3.4. Structural characteristics and properties of PP/BCSi composites

3.4.1. FTIR spectra

FTIR spectra of PP/BCSi samples are displayed in Figure 4. Figure 4a shows the specific absorption bands of PP, such

as the stretching vibrations (ν) of C-H bonds at 2838 - 2950cm^{-1} bending vibrations (δ) of CH_3 at 1454, δ(CH_2) at 1375cm^{-1}. There are pronounced peaks appearing at around 1163; 1085cm^{-1} and 795cm^{-1}, 777cm^{-1}, which are attributed to stretching (ν) and bending (δ) of Si-O (Si-O-C, Si-O-Si) in spectra of PP/BCSi [11, 12]. The presence of Si-O bonds on the FTIR of PP/BCSi samples confirms that PP/BCSi composite samples consist of PP and BCSi components, and these particles are dispersed into the PP resin.

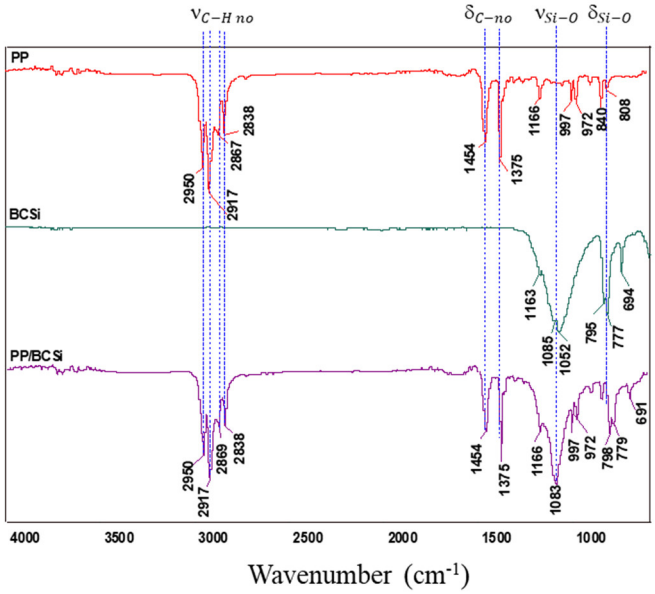
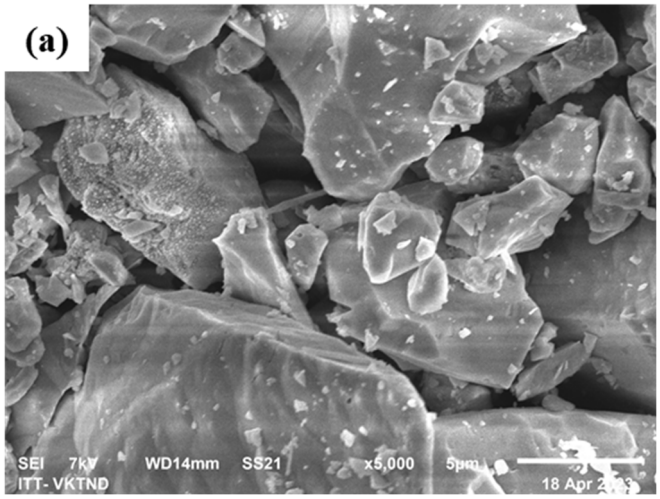


Figure 4. FTIR spectra of PP, BCSi and PP/BCSi composites

3.4.2. Scanning electron microscope (SEM)

Figure 5 shows the SEM images of the silica sand powder and PP/BCSi composite material. The BCSi particles are evenly distributed in the PP matrix and firmly attached to it, which is due to the positive interaction between the PP matrix and silica sand powder. There are no holes in the PP substrate material at the BCSi particle positions. This phenomenon can impact the mechanical properties of the materials, which will be discussed further in the next investigation.



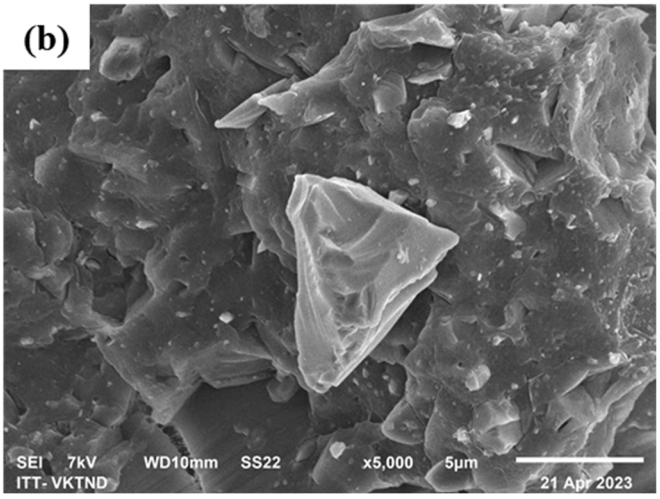


Figure 5. SEM images of (a) silica sand powder and (b) PP/BCSi composite material

3.4.3. Mechanical properties of PP/BCSi materials

To determine the difference in hardness between samples that used silica sand powder and did not by Shore D hardness method with a flat plate test sample that was 5mm thick and performed a hardness mapping test multiple times. The results of this test were used to create a hardness distribution chart, which can be seen in Figure 6.

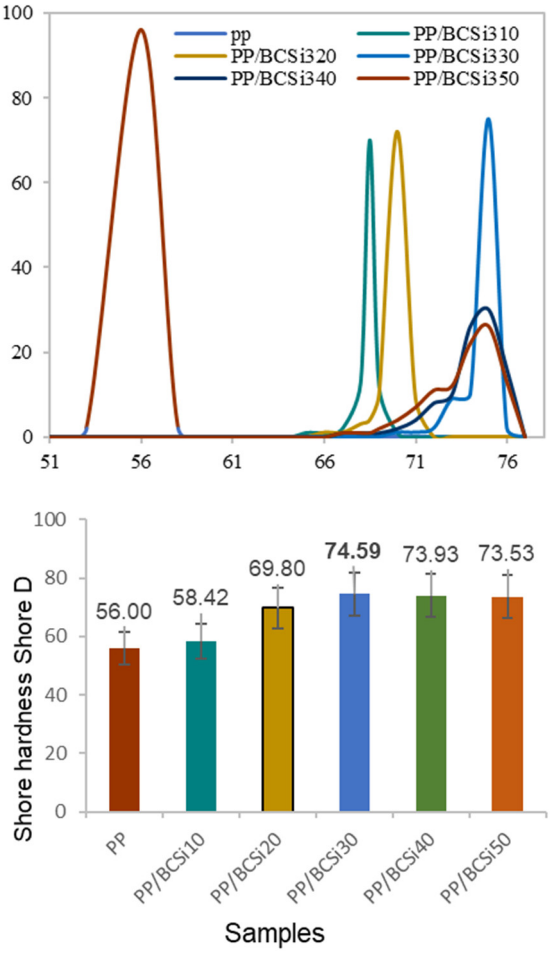


Figure 6. Shore D hardness of PP/BCSi composite materials

It can be clearly seen that the shore D hardness of the PP/BCSi composites is higher than that of the neat PP and increases from 56 to about 74.59 with increasing Silica sand powder contents from 10 to 30 wt.%. However, when contents becomes higher, with high concentration content lead to BCSi particle clusters, lowering in shore D of the composites but not significantly. Therefore, the sample with a 30% ratio has the best dispersion for the test mentioned above. This outcome is consistent with other mechanical properties of the material, including tensile strength and impact strength in Table 3.

Table 3. Mechanical properties of PP/BCSi 30

BCSi Content (%)	Impact strength (kJ/m^2)	Tensile strength (MPa)	Elongation at break (%)	Shore D hardness
30	27.83	24.00 ± 1.0	414.57 ± 2.3	74.59 ± 2.4

PP/BCSi sample possesses both high hardness and impact strength, making it suitable for producing products that require these technical properties, such as helmets, motorcycle helmets, or blender shells,…

4. CONCLUSION

PP/BCSi composite materials were prepared by the melt mixing method. The ratios used were 70:30 for PP and BCSi, and various conditions were tested. The FTIR spectra, mechanical properties, and morphology of the PP/BCSi composite samples have been analyzed.

PP/BCSi composite material fabricated at 190°C, 50rpm, and with a pressed pressure of 65bar yielded the best outcome. This composite material had a tensile strength of 24.00MPa, a Elongation at break of 414.57%, a impact strength of $27.83 kJ/m^2$, and a Shore D hardness of 74.59. The addition of PP/BCSi30 silica sand powder to the PP resin base evened out the distribution and enhanced its hardness (an increase of 33.20%) compared to the original PP under the same conditions.

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THÔNG TIN TÁC GIẢ

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