Eksergi
Jurnal Ilmiah Teknik Kimia
Vol 21, No. 3. 2024

ISSN: 1410-394X
e-ISSN: 2460-8203

Effects of Kaolin Powder and Borax Addition on Adhesion Properties of Soy Protein Isolate-Based Adhesives

Budhijanto*, Theresia Winona Rosari Widodo, Gilang Afif Setya Ramadhani

[a] *Chemical Engineering Department, Faculty of Engineering, Gadjah Mada University, Bulaksumur, Sleman, 55281, Indonesia*

Artikel histori :

Diterima 13 April 2024
Diterima dalam revisi 13 Agustus 2024
Diterima 17 Agustus 2024
Online 23 Agustus 2024

ABSTRAK: Sebuah strategi untuk meningkatkan sifat adhesi dan umur simpan perekat berbasis *Soy Protein Isolate* dengan biaya yang terjangkau dapat dilakukan dengan menggunakan kaolin bubuk sebagai *filler* dan boraks sebagai antijamur. *Bioadhesive* akan disintesis melalui denaturasi menggunakan *Sodium Lauryl Sulfate* (SLS), dilanjutkan dengan *crosslinking* menggunakan resin epoksi (ER) sebagai *crosslinker*, penambahan kaolin bubuk sebagai *filler*, dan penambahan boraks untuk mencegah tumbuhnya jamur. Beberapa pengujian seperti viskositas, *solid content*/kandungan padat, kuat geser kering, kuat geser basah, *FTIR Spectroscopy*, analisis SEM, serta umur simpan dilakukan untuk mengukur sifat adhesi perekat alami yang dihasilkan. Hasil penelitian menunjukkan bahwa kuat geser yang optimum, baik kering maupun basah diperoleh dengan menambahkan 1,5 wt% bubuk kaolin yang menghasilkan kuat geser sebesar 2,2684 MPa dan dengan penambahan 2 wt% boraks meningkatkan kuat geser hingga 2,3207 MPa. Penambahan boraks pada perekat juga terbukti dapat meningkatkan daya tahan terhadap air serta menghambat pertumbuhan jamur sehingga umur simpan dapat lebih lama.
Kata Kunci: *bioadhesive*; boraks; kaolin; *soy protein isolate*

ABSTRACT: A low-cost strategy was developed to improve the adhesion properties and long shelf life of soy protein isolate-based adhesive, by using kaolin powder as filler and borax as an antifungal. Soy protein bioadhesive will be synthesized through denaturation using Sodium Lauryl Sulfate, followed by crosslinking using epoxy resin as a crosslinker, kaolin powder addition as filler, and borax addition to inhibit the fungal growth. Some analysis such as viscosity, solid content, dry shear strength, wet shear strength, FTIR Spectroscopy, SEM analysis, and shelf life were done to measure the adhesion properties of the bioadhesive. The result shows that the best shear strength, both dry and wet was obtained by adding 1.5% w/w kaolin powder with shear strength resulted 2,2684 MPa and with addition 2% w/w borax increased the shear strength up to 2,3207 MPa. Borax addition had been proven in improving the water resistance of the adhesive also inhibit fungal growth resulting extended the shelf life of the adhesive.
Keywords: bioadhesive; borax; kaolin powder; soy protein isolate

1. Introduction

Adhesive is a material used to permanently glue an object through the gluing process (Ebnesajjad & Landrock, 2015). Adhesives play an important role in life, one of which is in the wood industry. In general, adhesives are made from petroleum-based chemicals, such as urea-formaldehyde or phenol-formaldehyde. These synthetic adhesives have strong adhesion, are easy to use, and are inexpensive (Koesman et al., 2023). However, the application of this synthetic adhesive in the production of wood composites can release free formaldehyde which can harm health and the environment because it is a toxic and carcinogenic material. In addition, formaldehyde also comes from non-renewable fossil resources and has limited reserves (Chen, 2007).

Bioadhesive is one solution to replace synthetic glue used in the market today (Wang, Zhao, et al., 2019). Soy protein isolated (SPI) is one of materials that can be used as bioadhesive. This material is made from soybeans which require a low cost to process, and the amount is still abundant (C. Liu et al., 2017). However, the adhesive produced from soybeans still has many disadvantages, such as relatively

* Corresponding Author
Email: budhijanto@ugm.ac.id

short life and poor water resistance (Cui et al., 2023). To create a better soybean-based glue, various methods have been done, such as adding stuffing or fillers, adding preservatives, and other chemicals (Nugroho et al., 2023).

Soy protein isolate is a natural ingredient that has a high enough protein content so that it can be used as a substitute for biobased adhesives (X. Liu et al., 2018). Several modifications have been developed to improve the adhesion properties and bond strength of this soybean-based adhesive, such as the addition of urea, Sodium Dodecyl Sulfate (SDS), and thermal-acid treatments (Syaichurrozi et al., 2016). However, soybean-based adhesives that have been developed so far have low water resistance and produce a high viscosity. The use of crosslinkers is also improve the quality of this soybean-based adhesive such as epoxy resin, polyamide-epichlorohydrin, and polyisocyanate (Ghahri et al., 2022). Epoxy resin can be an alternative as a crosslinker because the price is quite affordable compared to other materials.

Previous studies have stated that organic-inorganic hybridization polymer materials can improve the mechanical properties of adhesives (Akpinar et al., 2017). Several types of inorganic materials such as attapulgite and montmorillonite can be used to modify soybean-based adhesives. Kaolin powder can be used as an alternative material because it is low-cost compared to other materials (Patnode et al., 2022). As an important layered silicate mineral, kaolin has a highly reg snake sheet structure, good plasticity, easy dispersibility, high whiteness, large specific surface area, and excellent electrical insulation (Hermawan et al., 2021).

Another disadvantage of bioadhesives compared to synthetic glues that have been on the market so far is their short life. Natural materials tend to easily grow mold so the glue will break down in a fairly short period of time. The addition of an anti-fungal agent can be done to increase the durability of the adhesive (Zhang et al., 2022). One of the materials that can be used as an anti-fungal agent is borax. Borax itself is commonly used as a wood preservative in industry (Setyoningrum et al., 2018).

In this study, isolated soy protein (SPI) will be made into a bioadhesive by using SLS as a denaturant and epoxy resin (ER) as a crosslinker. The adhesive will be added with kaolin powder (KP) and borax which is expected to improve the quality of this natural soy-based adhesive. In addition, the addition of borax is also expected to increase the life of the adhesive by preventing the growth of fungi. In this research, condensation polymerization is done for soy protein adhesive production. Soy protein adhesive is formed by polycondensation reaction, where the amide monomer from SPI chemically bonds and formed longer chains of polyamide with high molecular weight. Therefore, this study will aim to determine the effect of adding these additives to the quality of the resulting bioadhesive, as well as finding the optimal amount for fine kaolin powder as a filler and borax as an antifungal agent.

2. Materials and Methods

Marksoy brand Soy Protein Isolate (SPI) was obtained from PD. Hamparan Rejeki, Indonesia. Sodium Lauryl Sulfate (SLS) was obtained from CV. Progo Mulyo, Indonesia. Aquadest, Borax, and wooden block were obtained from Chemical Reaction Laboratory, Chemical Engineering Department, Gadjah Mada University. Epoxy Resin (ER) was obtained from Nusakimia, Indonesia. Kaolin powder was obtained from Kimia Makmur, Indonesia.

2.1 Adhesive preparation

Soy Protein Isolated (SPI) was weighed as much as 15 grams and 85 mL of aquadest was added to a 250 mL beaker glass then stirred manually using a glass stirrer until a slurry was formed. After that, stirring is carried out continuously during the reaction using stirrer motor. The temperature is set on a scale of 55 – 60°C and the speed of stirring is adjusted accordingly. The SPI solution was heated for 5 minutes at a temperature of 55 – 60°C and the temperature was maintained constant. Thirty-five SLS was added to the sample and stirring was continued for 20 minutes at 80°C. Thirty five grams of SPI solution mixed with 100 grams of Epoxy Resin then stirred at room temperature (25 – 30°C).

Kaolin powder as much as 1.5 wt% was added to the SPI solution, then carried out and stirring was carried out for 10 minutes at room temperature. Sample preparation was repeated for powdered kaolin as much as 3 wt%, 4.5 wt%, 6 wt% and 7.5 wt%.

Borax as much as 1 wt% was added to the SPI solution, then carried out and stirring was carried out for 10 minutes at room temperature. Sample preparation was repeated for powdered kaolin as much as 2 wt%, 3 wt%, 4 wt% and 5 wt%. After the reaction is complete, the adhesive is put into the container to be cured for 1 day.

2.2 Viscosity measurement

After curing for a day, the adhesive will be tested for viscosity using a Brookfield Viscosimeter according to ASTM-D1084. As much as 100-150 mL of adhesive is put into the container, the selection of the spindle size and rotation speed depends on the viscosity of the adhesive. A suitable spindle is installed in the tool and the rotation speed is adjusted. Viscosity is recorded when the % torque has shown a figure of 10–90%.

2.3 Solid content measurement

The adhesive was weighed as much as 1 gram and placed on a petri dish. The oven is heated to a temperature of 100°C, then the adhesive is baked for 1 hour. After that, the sample was cooled in a desiccator for 10 minutes and the sample was weighed. The experiment was repeated 2 times, so that each sample had 3 solid content data.

2.4 Dry shear strength measurement

The adhesive was applied to a 3×3 cm wooden plank as much as ± 0.1 gram following the ASTM-D906 standard. Each sample was applied to 5 pairs of wood. The glued samples were subjected to a pressure load of 1,6868 N/mm^2

ISSN: 1410-394X
e-ISSN: 2460-8203

and allowed to stand at room temperature 25 – 30°C. After 1 day, the load was removed, and the sample was allowed to stand for 7 days before testing. Furthermore, the shear strength of the sample was tested using the Universal Testing Machine to apply a controlled shear force at a constant withdrawal speed of 10 mm/min until adhesive failure occurred. The test was conducted with the specimens carefully aligned between the machine's fixed and movable grips, ensuring that the load was applied uniformly along the specimen's axis, as per ASTM-D906 guidelines.

2.5 Wet shear strength measurement

The adhesive was applied to a 3×2.5 cm wooden plank as much as ± 0.1gram following the ASTM-D1183 standard. Each sample was applied to 5 pairs of wood. The glued samples were subjected to a pressure load of 1,6868 N/mm^2 and allowed to stand at room temperature 25 – 30°C. After 1 day, the load was removed, and the sample was allowed to stand for 7 days before testing. Before testing, the sample was immersed in water for 3 hours first and then dried for 1 hour. After that, the wet shear strength of the sample was tested using the Universal Testing Machine to apply a controlled shear force at a constant withdrawal speed of 10 mm/min until adhesive failure occurred. The test was conducted with the specimens carefully aligned between the machine's fixed and movable grips, ensuring that the load was applied uniformly along the specimen's axis, as per ASTM-D906 guidelines.

2.6 Fourier transform infrared (FTIR) spectroscopy

In the analysis of this test, an FT-IR Mb 3000 Spectrophotometer was used. The wavelengths used were between 4000 and 400 cm^{-1} with a resolution of 4 cm^{-1} and 32 scanning.

2.7 Scanning electron microscopy (SEM) analysis

In the analysis of this test, an electron microscope was used to obtain images of a sample by scanning the surface with a focused beam of electrons.

3. Results and Discussion

3.1. Reaction Mechanism

There are several reactions that occur, first reaction is protein denaturation with SLS, polymerization reaction with epoxy resin, and the addition of filler to the adhesive. Initially, SPI undergoes denaturation through the action of SLS, as depicted in Figure 1. During this process, the hydrocarbon tails of SLS bind to the protein chains, causing the protein structure to unfold. The SLS molecules coat the protein with a negative charge, rendering it more reactive and ready to bind with other chemical groups (Ristianingsih, 2021). Denatured proteins will undergo condensation polymerization with epoxy resins to form polyamides. The amine group in the protein will release hydrogen ions which will then react with the hydroxyl group of the epoxy resin (Akmalina, 2019). Proteins and epoxy resins that have released their respective groups will bond to each other to form polyamides. This is a critical step where the protein and epoxy resin, having released their respective functional groups, bond to form a new polymeric structure, as shown in Figure 2. The resulting bioadhesive still has relatively weak water resistance (Luo et al., 2016).

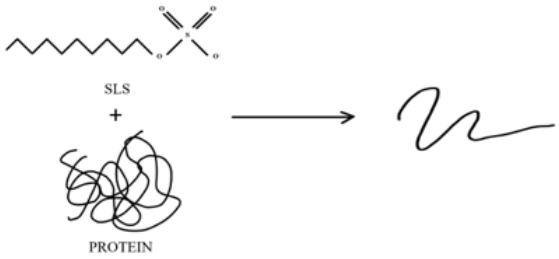

Figure 1. Protein Denaturation Process

The reaction that occurs next is the addition of filler to the adhesive through organic-inorganic hybridization between SPI, epoxy resin, and kaolin powder as shown in Figure 2. The cyclic bond in the epoxy resin will open so that it can react with the -OH and -NH2 groups in SPI and -OH group in kaolin. This bioadhesive still has the possibility of being exposed to microbial attack so it has a limited shelf life (Larregle et al., 2021). Therefore, the adhesive is then added borax as an antifungal to increase shelf life.

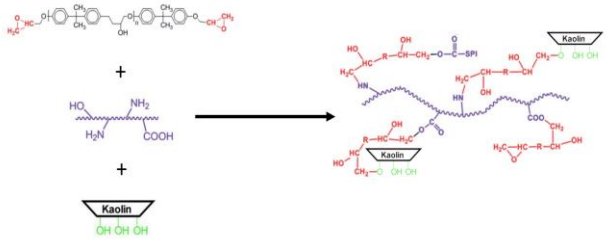

Figure 2. Reaction between SPI, Epoxy Resin, and Kaolin Powder

3.2. Shear Strength Analysis

Shear strength analysis was carried out according to ASTM-D906 standard for dry shear strength and ASTM-D1183 for wet shear strength. This test is carried out by testing the bioadhesive that has been applied to wood with an application area of 25 mm x 30 mm. The test was carried out by taking 5 data so that for each bioadhesive made it was applied to 5 pairs of wood. The five samples are treated equally to produce accurate data.

In the shear strength test, the dry shear strength for the blank solution was 1.3661 MPa and the wet shear strength was 1.2652 MPa. Based on the data from the shear strength listed in the graph in Figure 3, the optimum amount of kaolin

powder added is 1.5 wt%, with a dry shear strength value of 2.2684 MPa and a wet shear strength of 1.8832 MPa.

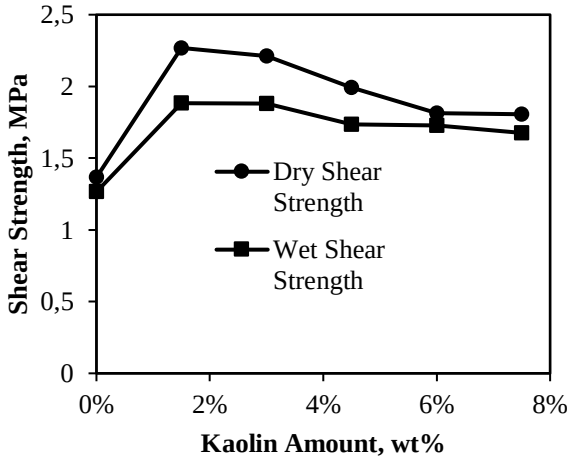

Figure 3. Dry Shear Strength and Wet Shear Strength for Each Kaolin Powder Percentage

The optimum amount of kaolin powder added to the adhesive is then added with various variations of borax. In the graph in Figure 4, the optimum amount of borax added to the adhesive is 2 wt% with a dry shear strength value of 2.3207 MPa and a wet shear strength of 2.1264 MPa. Based on the data obtained, the value of the wet shear strength tends to be lower than the dry shear strength because water does not have adhesive properties so it can reduce the shear strength value of the sample.

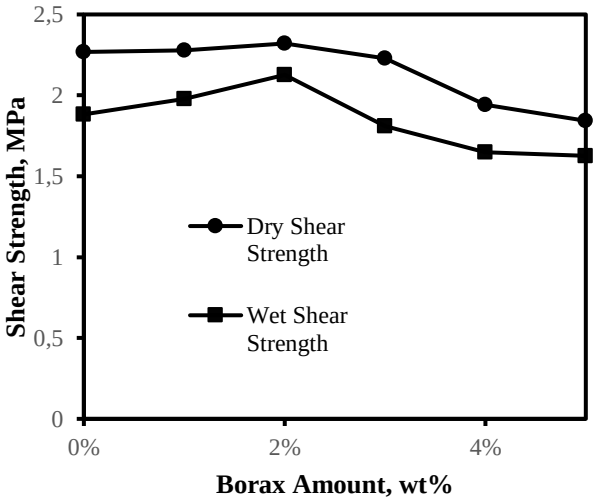

Figure 4. Dry Shear Strength and Wet Shear Strength for Each Kaolin Powder Percentage

There was an increase in shear strength both wet and dry from the blank sample when compared to SPI/K. This happens because in SPI/K, kaolin which functions as a filler will form hybridization which forms a tighter adhesive layer and increases the water resistance of the adhesive which is better. However, the more kaolin added, the dry and wet shear strengths decreased because too much kaolin particles tended to form clumps of kaolin solids which formed pores in the adhesive polymer produced. The pores formed increase the possibility of water penetrating the adhesive layer (Zhang et al., 2019). For the SPI/K/B samples, an increase in shear strength was obtained compared to SPI/K, especially in the wet shear strength. The borax will strengthen the polyamide bonds that are formed. However, the addition of excess borax will reduce the resulting shear strength because borax will make the adhesive agglomerate more, which is caused by the diffusion of Na^+ ions which can result in a decrease in the repulsion of the protein structure so that the stretched structure curls up (Xu et al., 2014).

To further contextualize the performance of the developed bioadhesive, it is important to compare its shear strength with that of commercially available wood adhesives. Commercial products such as polyvinyl acetate (PVA) or polyurethane-based adhesives typically exhibit dry shear strengths ranging from 2.5 MPa to 4 MPa, and wet shear strengths ranging from 1.5 MPa to 3 Mpa (Wang, Zhu, et al., 2019).

The developed bioadhesive, with its dry shear strength of 2.3207 MPa and wet shear strength of 2.1264 MPa, demonstrates comparable performance to these commercial adhesives. Notably, the bioadhesive's wet shear strength is competitive, suggesting it could serve as a viable, sustainable alternative in applications where resistance to moisture is crucial.

3.3. Viscosity Analysis

The viscosity of the sample tested was quite thick, so spindle 6 was used. The viscosity of the SPI/K sample varied from 10,740-31,390 cP as shown in Figure 5. From the data obtained, the more kaolin powder added, the viscosity of the sample will increase. Based on the shear strength analysis data, it is known that the optimum amount of kaolin added at 1.5 wt% has a viscosity of 12,580 cP. Meanwhile, the viscosity of the sample with the addition of borax also experienced an increase in viscosity as more borax was added to the adhesive as shown in Figure 6. The optimum viscosity of the sample with 2% borax is 28,336 cP. The more solids added to the adhesive, the higher the viscosity the adhesive will have.

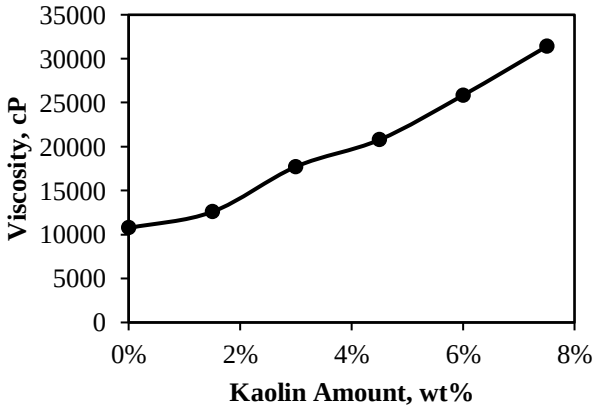

Figure 5. Viscosity for Each Kaolin Powder Amount

ISSN: 1410-394X
e-ISSN: 2460-8203

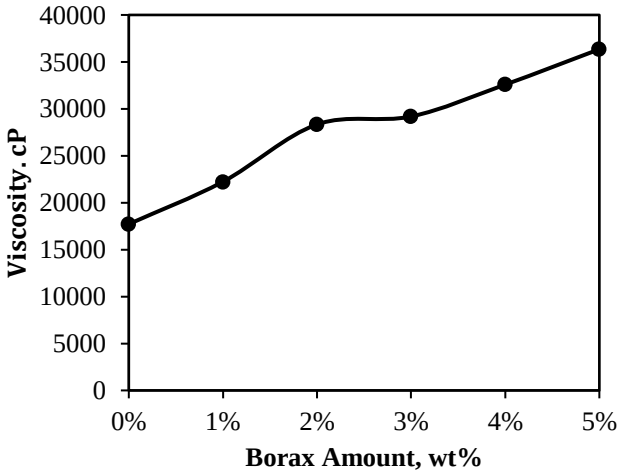

Figure 6. Viscosity for Each Borax Amount

3.4. Solid Content Analysis

Based on the solid content test in Figure 7, the data obtained for the blank sample was 24.17%. For the SPI/K sample, the solid content value increased little by little from 26.61 – 31.22%. There was a significant increase in solid content from samples without kaolin to samples with 3 wt% added kaolin. For the SPI/K/B sample with a kaolin content of 1.5 wt%, there was a significant increase from the samples that did not add borax to the samples that added 1% borax.

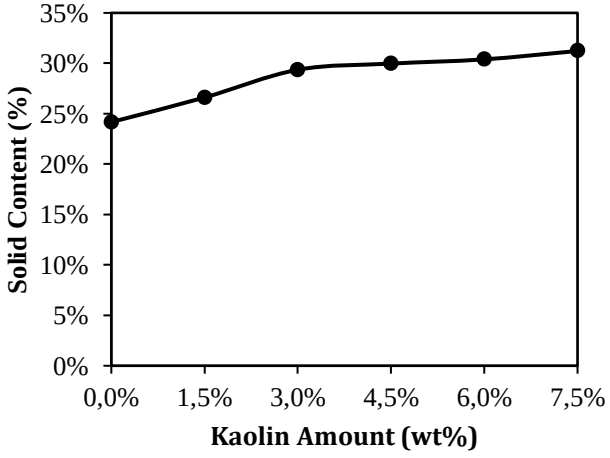

Figure 7. Solid Content for Each Kaolin Amount

Meanwhile, the increase in solid content in various variations of adding borax increased but not too significantly in the range of 34.29 – 39.46%. The increasing number of solids, both kaolin and borax in the adhesive causes the solid content value to also increase as depicted in Figure 8. The addition of borax to the adhesive produces a solid content that complies with SPI-based adhesive standards in the range of 30-40% (Tabarsa et al., 2011).

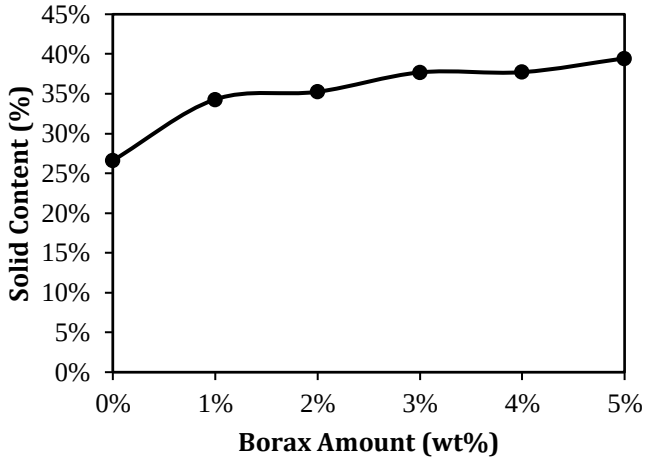

Figure 8. Solid Content for Each Borax Amount

3.5. FTIR (Fourier Transform Infrared) Spectroscopy Analysis

FTIR testing was carried out for blank, optimum kaolin, and optimum borax samples as shown in Figure 9. There is a C=O stretch bond, marked by a peak at a wavelength of 1,050 cm^{-1} at SPI/K and SPI/K/B which is produced by the bond between kaolin and epoxy resins. At a wavelength of 1270-1230 cm^{-1} there is a peak which indicates that there is a C-O-C stretch bond produced by the SPI bond and the epoxy resin. The most significant difference seen from the three samples is the peak height which lies in the wavelength range of 3,200 – 3,600 cm^{-1} (OH group). The highest peak is owned by the SPI/K/B sample with the lowest transmittance. This increasing number of OH bonds is produced by the opening of cyclic bonds in the epoxy resin which binds to the O atoms of SPI and kaolin.

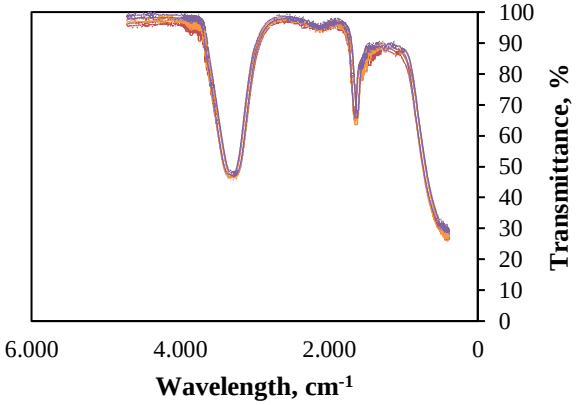

Figure 9. FTIR Result

3.6. SEM (Scanning Electron Microscopy) Analysis

SEM testing was carried out using blank samples (REF), optimum kaolin (SPI/KP), and optimum borax (SPI/KP/B) as shown in Figure 10.

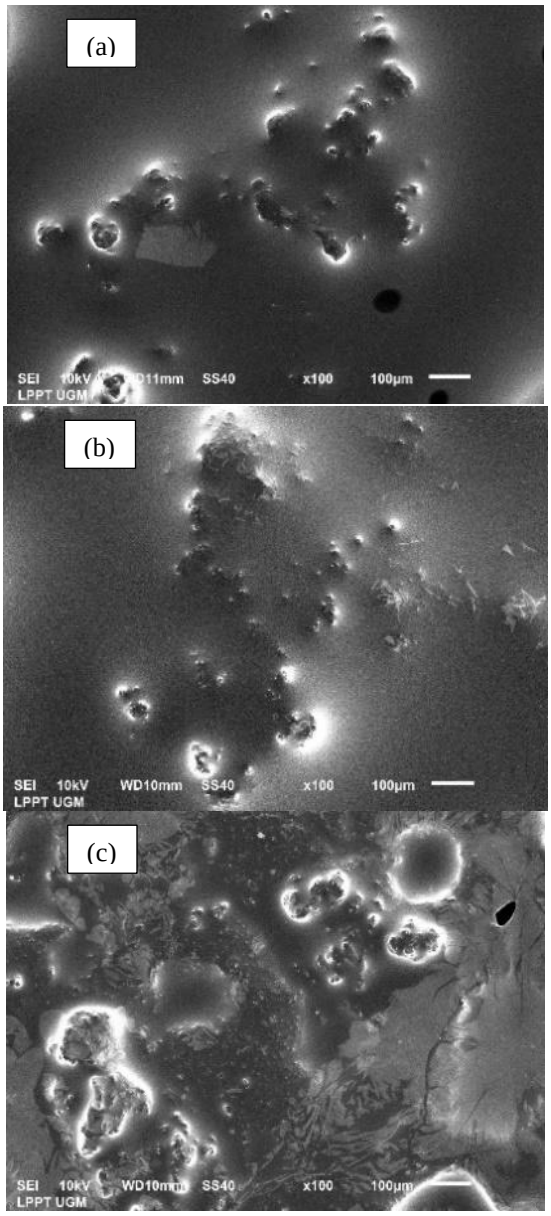

Figure 10. SEM Result (a)REF (b)SPI/KP (c)SPI/KP/B

Figure 10(a) represents the SEM image of the blank sample (REF). The surface of the REF has parts of the pores that are less filled even though they look quite homogeneous. Figure 10(b) shows the SPI/KP sample, where the surface of the SPI/KP sample looks denser. This indicates that kaolin can work as a filler in the adhesive. The SPI/KP sample has a stronger shear strength than REF, due to the denser adhesive structure. Figure 10(c) illustrates the SPI/KP/B sample, which shows larger lumps than REF or SPI/KP. The addition of borax to the adhesive makes the adhesive thicker so that the viscosity increases, and over time, borax will tend to make the adhesive thicker and form lumps even though the adhesive will last longer.

3.7. Fungal Growth Analysis

The adhesive sample that has been made is stored in a tightly closed container to prevent contact with air, which can trigger mold growth. The samples observed were SPI/KP/B with an optimum amount of 1.5 wt% kaolin and 2 wt% optimum borax. During observation on the 60th day after making the adhesive, there was still no visible fungal growth at all. As shown in the SEM image of SPI/KP/B samples in Figure 11, the surface of the SPI/KP/B sample remains clear and smooth, with no evidence of fungal structures or contamination even after 60 days. This SEM result further supports the effectiveness of borax in preventing mold growth on natural adhesives, demonstrating its role as a reliable antifungal agent in maintaining the integrity of the bioadhesive over time.

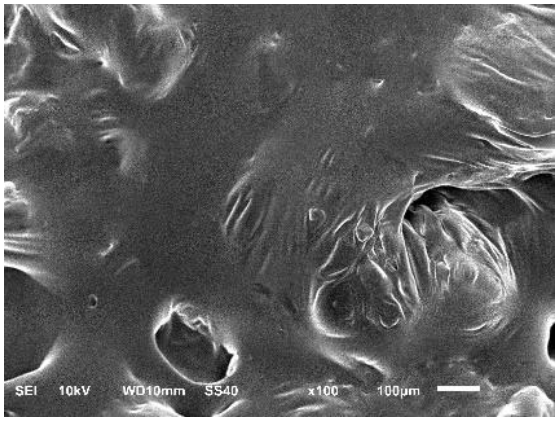

Figure 11. SEM Result SPI/KP/B After 60th days

4. Conclusion

The conclusion that can be drawn from this study is that the addition of filler in the form of kaolin can increase the shear strength of the bioadhesive, with an optimum amount of 1.5 wt%. The addition of anti-fungal in the form of borax further enhances the shear strength, particularly under wet conditions, with an optimum amount of 2 wt%. The viscosity of the bioadhesive also increased with the addition of kaolin and borax, although not significantly. The solid content tended to increase with the inclusion of these additives, and while borax extended the shelf life of the adhesive, it also led to increased thickening and clotting over time. The resulting optimum dry shear strength is 2.3207 MPa and the resulting optimum wet shear strength is 2.1264 MPa. When compared to commercially available wood adhesives, such as polyvinyl acetate (PVA) and polyurethane-based adhesives, which typically exhibit dry shear strengths between 2.5 MPa and 4 MPa, and wet shear strengths between 1.5 MPa and 3 MPa, the developed bioadhesive demonstrates competitive performance. This suggests that the bioadhesive developed in this study not only offers a sustainable alternative but also achieves shear strength values that are comparable to those of current market products.

Acknowledgement

The authors are grateful for the financial support from the Chemical Engineering Department, Gadjah Mada University.

ISSN: 1410-394X
e-ISSN: 2460-8203

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