Menara Perkebunan 2024, 92(1), 82-89
p-ISSN: 0125-9318/ e-ISSN: 1858-3768
http://dx.doi.org/10.22302/iribb.jur.mp.v92i1.566
Accreditation Number: 164/E/KPT/2021

Fabrication and characterization of biocomposite pellets from cassava starch and oil palm empty fruit bunch fibers

Fabrikasi dan karakterisasi pelet biokomposit dari pati singkong dan serat tandan kosong kelapa sawit

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Received 28 Jan 2024 / Revised 28 Mar 2024/ Accepted 26 Apr 2024

Abstrak

Tandan kosong kelapa sawit (TKKS) merupakan biomassa lignoselulosa yang dapat dimanfaatkan untuk menghasilkan biokomposit sebagai alternatif pengganti bahan tidak terbarukan, seperti plastik. Umumnya, produksi biokomposit menggunakan TKKS yang sudah diolah menjadi selulosa, mikrokristalin selulosa, atau nano selulosa, yang dicampurkan dengan pati. Namun, proses preparasi TKKS menjadi beragam jenis selulosa tersebut memerlukan proses yang panjang dan banyak bahan kimia. Preparasi TKKS yang lebih sederhana, seperti pemotongan menjadi serat yang lebih pendek dan tanpa penggunaan bahan kimia, diharapkan dapat mempersingkat proses. Penelitian ini bertujuan untuk memproduksi dan mengevaluasi karakteristik pelet biokomposit dari kombinasi pati singkong dan serat TKKS. Serat TKKS pendek (berukuran 3-5 mm) dengan variasi konsentrasi yaitu 0, 5, 10, 15, dan 20% ditambahkan ke dalam pati singkong sebelum proses pencampuran dengan bahan-bahan lainnya. Ekstruder sekrup kembar yang digunakan untuk membuat pelet biokomposit diatur pada enam zona suhu antara 85-140 °C dan kecepatan sekrup pada kisaran 160-190 rpm. Hasil penelitian menunjukkan bahwa serat TKKS dengan konsentrasi yang lebih tinggi menghasilkan pelet yang berwarna semakin gelap cenderung keabu-abuan. Pelet biokomposit mempunyai massa jenis 1,322-1,417 g cm^{-3}*. Hasil SEM menunjukkan terdapat aglomerasi pada permukaan pelet. Kelarutan pelet biokomposit dalam air berkisar antara 32,97 – 36,44 %. Sebagai kesimpulan, pelet biokomposit dapat dibuat dari campuran antara pati singkong dan serat TKKS hingga 20%. Dalam aplikasinya untuk produksi kemasan kaku, performa pelet biokomposit dapat ditingkatkan dengan mencampurkannya dengan bijih poli propilen daur ulang.*

[Kata kunci: pati singkong, compounding, massa jenis, ekstruder sekrup ganda]

Abstract

Oil palm empty fruit bunches (OPEFB) are lignocellulosic biomass that can be used to produce a biocomposite as an alternative to substitute non-renewable materials, such as plastic. Generally, the production of biocomposites uses OPEFB, which has been processed into cellulose, microcrystalline cellulose, or nanocellulose and is mixed with starch. However, the OPEFB pretreatment into various types of cellulose requires a long process and many chemicals. The OPEFB pretreatment with less process, such as cutting to shorter fibers and without chemicals, was expected to shorten the process. This study aims to produce and evaluate the characteristics of biocomposite pellets from a combination of cassava starch and OPEFB fibers. Short OPEFB fibers (3-5 mm) with varying concentrations of 0, 5, 10, 15, and 20% were added to the cassava starch before mixing with other materials. The twin screw extruder used to produce biocomposite pellets was set at six temperature zones ranging from 85-140 °C and the screw speed in the range of 160-190 rpm. The results show that higher concentrations of OPEFB fibers produced darker pellets, which tended to be greyish. The biocomposite pellets had densities of 1.322-1.417 g cm^{-3}. SEM results show some agglomerations on the surface of starch-OPEFB fibers biocomposite pellets. The water solubility of biocomposite pellets ranged from 32.97 – 36.44%. In conclusion, biocomposite pellets could be produced from a mixture of cassava starch and OPEFB fibers up to 20%. In its application for rigid packaging production, the biocomposite pellets' performance could be improved by mixing them with recycled polypropylene.

[Keywords: cassava starch, compounding, density, twin screw extruder]

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How to Cite: Faramitha, Y., Dimawarnita, F., Cifriadi, A., Widiastuti, H., & Herawan, T. (2024). Fabrication and characterization of biocomposite pellets from cassava starch and oil palm empty fruit bunch fibers. *Menara Perkebunan, 92*(1), 82-89
http://dx.doi.org/10.22302/iribb.jur.mp.v92i1.566

Introduction

As the biggest crude palm oil (CPO) producer worldwide, Indonesia produces large amounts of biomass from the palm oil industry activities. These biomasses include empty fruit bunch (EFB), palm kernel shell, mesocarp fiber, and palm oil mill effluent (POME) (Hambali & Rivai, 2017). Oil palm empty fruit bunch (OPEFB) is the major solid biomass, with 23% of EFBs produced from processed fresh fruit bunches (FFB) (Derman et al., 2018). The utilization of OPEFB is urgently needed in order to manage this abundant by-product so as not to become waste. In oil palm plantations, OPEFBs are conventionally used as mulch material and fertilizers, as OPEFBs contain nutrients that could improve soil properties (Sari et al., 2022). Besides, advanced OPEFB utilizations, such as biofuels and materials, have also been studied in the last decades (Chang, 2014; Cheng et al., 2019). Research in OPEFB utilization is in high demand because there are many opportunities to develop this biomass into valuable products.

The OPEFB, as an abundant lignocellulose source, is potentially developed to be biocomposite as an alternative to substitute non-renewable and non-degradable materials, like plastics. Biocomposite is an eco-friendly composite that can be formed from a combination of a matrix, like natural polymers, and reinforcement from natural fibers (Bharath & Basavarajappa, 2016). Starch blend-based bioplastics that use natural fiber as reinforcement are also grouped as biocomposite. The development of biodegradable composites from agro-waste for application in the packaging sector has been a great breakthrough (Bhardwaj et al., 2020). The use of OPEFB as biocomposite material offers several advantages, including abundant and cheap material, strong fiber properties with low density, versatile product use, increasing the economic value of OPEFB, and contribution to sustainable waste management (Padzil et al., 2020; Rao & Ramakrishna, 2022). This biocomposite can be used as an intermediate product to produce eco-friendly products, which recently gained high social interest and demand (Gajula et al., 2019).

As a reinforcement agent in starch blend biocomposites, the OPEFB is commonly treated to obtain cellulose (Isroi et al., 2017). Furthermore, a number of studies have been focused on the conversion of EFB cellulose into microcrystalline cellulose (MCC) and nanocrystalline cellulose (NCC) and their application as reinforcement in starch biocomposite (Hairani et al., 2015; Perera et al., 2022). The use of cellulose, MCC, or NCC is expected to improve the compatibility between OPEFB fiber derivatives and starch, thus improving the performance of biocomposites. However, the pretreatment of raw material, OPEFB, into cellulose and further into MCC and NCC are complex, requiring many physical and chemical treatments. This study focused on using less processed OPEFB, in which OPEFB fibers were incorporated with the starch matrix as materials for biocomposite. The OPEFB still contains lignin, hemicellulose, and cellulose and is not subjected to further chemical treatment. Based on our knowledge, studies on biocomposite fabrication from raw OPEFB fibers and starch are still limited. Besides, research on developing biocomposites based on starch mixtures is generally limited to laboratory scale using the casting method. This method is considered less applicable to the industrial scale. Producing biocomposite pellets using the twin screw extrusion method is considered appropriate for industrial-scale production. This research aims to produce and characterize biocomposite pellets from a combination of cassava starch and OPEFB fibers as fillers using the twin screw extrusion method.

Materials & Methods

Materials

The cassava starch used was produced by PT. Budi Starch & Sweetener Tbk (Indonesia) and the analysis showed that this cassava starch has a water content of 9.79%. The OPEFB was obtained from PTPN VIII Cikasungka-Banten Plantation. Glycerol and sorbitol with technical grade quality were purchased from PT. Ganendra Global Labtekindo (Indonesia).

Preparation and characterization of OPEFB fibers

The dried OPEFBs were roughly chopped into long fibers using a machete. The long fibers of OPEFB were then chopped to 3-5 mm size using a chopping machine. The chopped OPEFB was filtered using a 100-mesh sieve to get a homogenous size of short OPEFB fibers. The OPEFB fibers were characterized for lignin, hemicellulose, alpha-cellulose, and water content. The lignin, hemicellulose, and alpha-cellulose content of OPEFB fibers was analyzed at Balai Besar Standarisasi dan Pelayanan Jasa Industri Selulosa (Bandung, Indonesia). Lignin content was measured according to SNI 8429, hemicellulose content was measured according to SNI 01-1561, and alpha-cellulose was measured according to ASTM D 1103. The water content of OPEFB fibers was calculated using the gravimetric method.

Fabrication of biocomposite pellets

Composite pellets were prepared by dry-mixing cassava starch, OPEFB fibers at different concentrations (0, 5, 10, 15, and 20 wt.%), and titanium dioxide for 5 minutes. The mixing temperature was maintained below 40 °C. Next, liquid ingredients such as 2% acetic acid, glycerol, and sorbitol at a ratio of 0.25: 1: 2 (v/v/v) were gradually added to the dry mixture while stirring until the mixture was evenly mixed. The final mixture was then extruded by using a co-rotating twin screw extruder (Nanjing Kerke Extrusion Equipment Co.Ltd., Cina). The six zones of heating section temperatures were set at 85-140 °C while the host speed was at 160-190 rpm. The extrudate formed was dried at room temperature for 24-48 hours and then cut into pellets (0.5 cm). Composite pellets were stored at room temperature in a closed tight container. Each treatment was repeated three times. The characterization of biocomposite pellets from each treatment included density, Field Emission – Scanning Electron Microscopy (FE-SEM), water content, and water solubility.

Density analysis

Density analysis was conducted according to ISO 1183-1 about the immersion method.

Characterization of Field Emission – Scanning Electron Microscopy (FE-SEM)

The morphology image of biocomposite pellets was analyzed by using scanning electron microscopy (SEM, JEOL JIB-4610F) operating at 1 kV.

Water content and solubility analysis

Water content was measured by the gravimetric method. One gram of biocomposite pellets was dried in the oven at 105 °C for 3 hours to remove the water. The sample was reweighed and dried at 105 °C for 1 hour to ensure the final weight was constant. Water content was measured by the following formula:

$$Water\ content\ (\%) = \left[\frac{W_w - W_d}{W_w}\right] x\ 100 \ldots\ldots\ldots [1]$$

W_w = weight of composite pellets before drying process (g)
W_d = weight of dried composite pellets (g)

The water solubility test was carried out by adopting a method from Marichelvam et al. (2019). A 2 g composite pellets in 100 mL deionized water were stirred at 180 rpm for 6 hours. Then, the composite pellets were filtered and dried in the oven at 70 °C for 24 hours until the final weight was fixed. The solubility percentage of soluble matter in composite pellets was determined by this following formula:

$$WS\ (\%) = \left[\frac{W_0 - W_f}{W_0}\right] x\ 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots [2]$$

WS = solubility in water (%)
W_0 = weight of composite pellets before immersion (g)
W_f = final weight of composite pellets after immersion (g)

Results & Discussion

Figure 1 presents the OPEFB fibers that had been chopped and filtered. The lignin, hemicellulose, α-cellulose, and water contents of these OPEFB fibers are 27.78, 24.81, 37.07, and 10.80 %, respectively (Table 1). Rao & Ramakrishna (2022) summarized the lignin, hemicellulose, and cellulose contents in OPEB, which were reported to range from 10.74 to 24.5 %, 19.15 to 35.87 %, and 23.70 to 65%, respectively. Cellulose becomes the major component in the OPEFB fibers, which comes after hemicellulose and lignin. Cellulose itself as a biomaterial is an attractive source as the crystallization degree and the microfibril formation can be adjusted through the treatment condition (Diyanilla et al., 2020). However, the lignin content of OPEFB may affect its use as a biomaterial because lignin is a complex heteropolymer that is difficult to degrade.

Figure 1. The fibers of oil palm empty fruit bunches (OPEFB)
Gambar 1. Serat tandan kosong kelapa sawit (TKKS)

Biocomposite pellets from all variation formulations are displayed in Figure 2. The loading percentages of OPEFB on starch-based composite formulation affected the color and texture of the resulting biocomposite pellets. The higher the loading percentages of OPEFB fibers, the darker the pellets' color and the more visible the OPEFB fibers in the pellets. The CS pellets without the reinforcement of OPEFB fibers had a beige color, while the composite pellets of starch and OPEFB fibers at 5 to 15 % tended to have color in the grey range. The biocomposite pellet had a blackish-brown color at the 20% loading of OPEFB fibers. This color difference was mainly influenced by the percentages of the OPEFB fibers, which passed through several heating zones in the twin screw extruder, thus making the biocomposite pellets became darker.

The density of starch-OPEFB composite pellets is provided in Figure 3. The highest density, 1.417 g cm^{-3}, was from cassava starch pellet (CS). The higher the OPEFB fiber content in the pellet, the lower the density of the biocomposite pellets. The CS-OPEFB 5% had a density of 1.403 g cm^{-3}, while CS-OPEFB 10% and 15% had similar densities at around 1.369-1.370 g cm^{-3}. The addition of 20% OPEFB fibers with cassava starch (CS-OPEFB 20%) produced the lowest density among others, 1.322 g cm^{-3}. A slight decrease in the density of biocomposites might be due to the low density of OPEFB compared to starch. According to Solikhin et al. (2016), the OPEFB structure that generally consists of stalks (20-25 %) and spikelets (75-80 %) had a density for stalks of 0.5 g cm^{-3}, while spikelet fiber ranges from 0.71 to 1.53 g cm^{-3}. Abdullah et al. (2019) reported that the density of cassava starch-based bioplastics without reinforcement was 1.3 g cm^{-3}.

The surface morphologies of biocomposite pellets with different loading percentages of OPEFB fibers are displayed in Figure 4. The surface of CS pellets without the addition of OPEFB fibers was relatively smooth and homogenous. The surface with few aggregates appeared at CS-OPEFB 5% pellets. Higher loading percentages of OPEFB fibers (≥10%) led to agglomeration structures, which means that OPEFB fibers were not dispersed uniformly in cassava starch-based composites.

Table 1. The content of lignin, hemicellulose, and alpha cellulose of OPEFB

Tabel 1. Kandungan lignin, hemiselulosa, dan selulosa alfa pada TKKS

Parameter *Parameter*	OPEFB *TKKS*
Lignin (%) *Lignin (%)*	27.78
Hemicellulose (%) *Hemiselulosa (%)*	24.81
α – cellulose (%) *Selulosa alfa (%)*	37.07
Water content (%) *Kandungan air (%)*	10.80

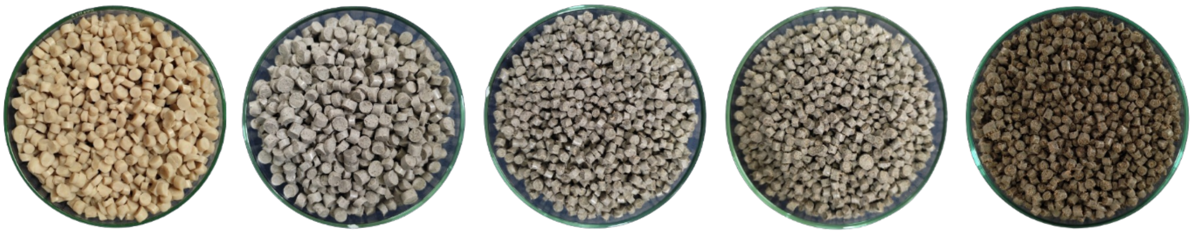
(a) **(b)** **(c)** **(d)** **(e)**

Figure 2. Biocomposite pellets from a mixture of starch with varying OPEFB fiber concentrations. (a) 0%; (b) 5%; (c) 10%; (d) 15%; (e) 20%

Gambar 2. Pelet biokomposit dari campuran pati dengan konsentrasi TKKS bervariasi. (a) 0%; (b) 5%; (c) 10%; (d) 15%; (e) 20%

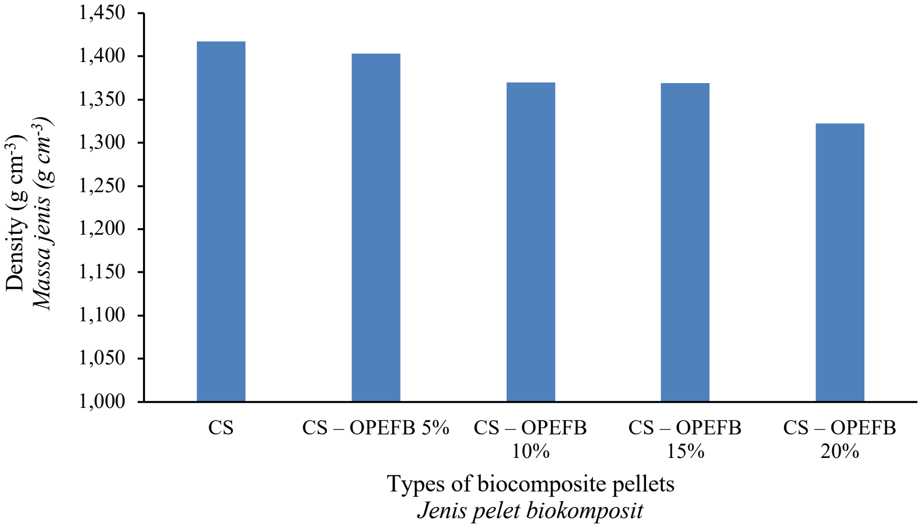

Figure 3. Density of biocomposite pellets from the mixture of starch with varying OPEFB concentrations. CS = Cassava starch and OPEFB = oil palm empty fruit bunch

Gambar 3. Massa Jenis pellet biokomposit dari campuran pati dengan konsentrasi TKKS bervariasi. PS = pati singkong; TKKS = tandan kosong kelapa sawit

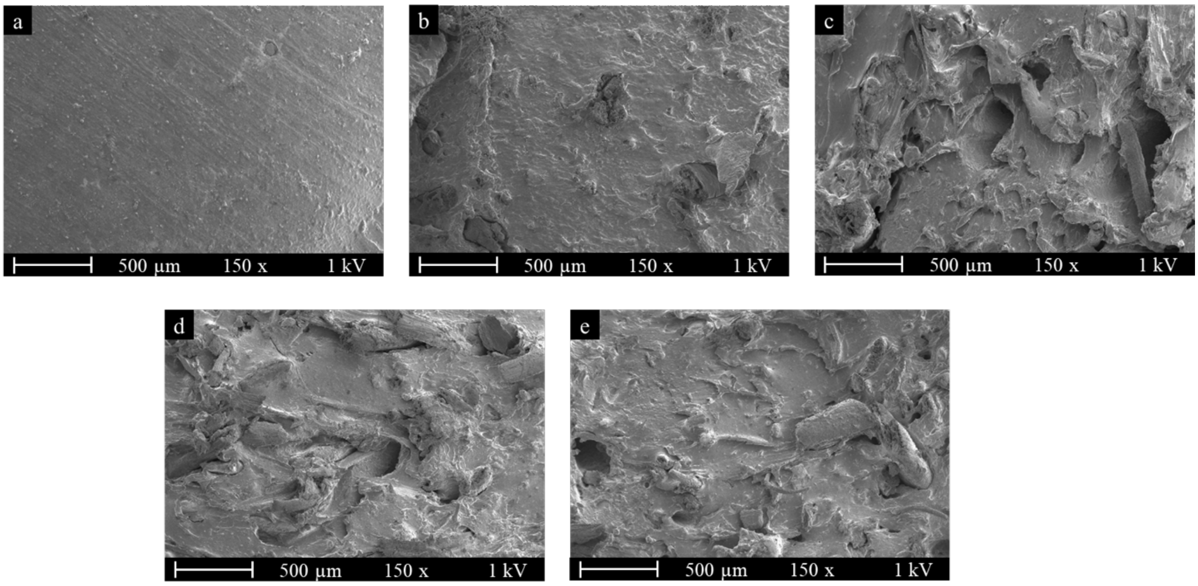

Figure 4. Field Emission – Scanning Electron Microscopy (FE-SEM) of biocomposite pellets from a mixture of starch with varying OPEFB concentrations. (a) 0%; (b) 5%; (c) 10%; (d) 15%; (e) 20%

Gambar 4. Field Emission – Scanning Electron Microscopy *(FE-SEM) pelet biokomposit dari campuran pati dengan konsentrasi TKKS bervariasi. (a) 0%; (b) 5%; (c) 10%; (d) 15%; (e) 20%*

The characteristics of OPEFB fibers used in this study immensely influence the compatibility between starch and OPEFB fibers. Compared to cellulose, microcrystalline cellulose, and nano cellulose as reinforcements, the OPEFB fibers were not treated with chemical modifications, so they might contain many impurities (such as waxes, silica, and fatty substances) that hinder the fiber-matrix interaction. Besides, the particle size of OPEFB fibers was bigger than that of treated fibers. A study by Yang et al. (2021) showed the difference in the surface berween raw and treated OPEFB fibers; many tyloses (round particles and small holes) were shown on the surface of raw OPEFB fibers, which were the impurities in the fibers. In contrast, the chemical-treated OPEFB fibers had a smooth surface as the impurities had been removed. Furthermore, the SEM results of starch-treated OPEFB fibers biocomposite present no fiber aggregate seen on biocomposite with the addition of treated OPEFB fibers up to 5%. Besides, the formation of starch granules might be caused by a decrease in hydrogen bonding interaction between plasticizers and starch due to the interaction of plasticizers with fibers (El Miri et al., 2015).

The water content of biocomposite pellets is presented in Figure 5, while their solubility in water is displayed in Figure 6. Overall, the biocomposite pellets loaded with OPEFB fibers had higher water content and solubility compared to the CS pellet. CS pellet had the lowest water content and solubility percentage of 9.94 and 32.97 %, respectively. At 5% loading of OPEFB fibers (CS-OPEFB 5%), the biocomposite pellet's water content and solubility percentage rose to 12.03 and 34.98 %, respectively. However, the water content and solubility decreased to 11.47 and 33.96 % at CS-OPEFB 10%, then at CS-OPEFB 15%, there were increases to 11.77 and 35.59 %, respectively. Biocomposite pellets with the highest water content and water solubility, 13.85 % and 36.44 %, were CS-OPEFB 20% pellets.

This study shows that water or moisture in the biocomposite pellet seemed to affect its water solubility, as the water solubility graph has a similar pattern with water content. The higher water contents and solubilities in biocomposite pellets loaded with OPEFB fibers were due to the natural properties of OPEFB fibers, which are hydrophilic. The hydrophilicity of natural fibers is attributed to the presence of hydroxyl groups, which are known to attract water molecules through hydrogen bonding (Hassan et al., 2010). Hanan et al. (2020) found that composites with higher OPEFB fiber content had higher water absorption. Since OPEFB fibers are prone to water/moisture, they might cause poor mechanical interlocking between cassava starch and OPEFB fibers. As shown in Figure 4, the SEM images display the presence of agglomeration (holes), thus allowing the water to enter from these surface porosities, which might cause the starch or the OPEFB fibers to dissolve or lose to a greater extent. Hence, the loading percentage of fibers could affect the water content and solubility of biocomposite pellets.

The compatibility between starch and fiber type influences the biocomposites' water solubility. Some modifications on the fibers, such as chemical treatment, could improve the compatibility and, thus, the performance of biocomposite pellets (Hassan et al., 2010; Nagarajan et al., 2021). Yang et al. (2021) used treated OPEFB fibers (removed impurities and lignin) and starch for composite materials, and the water solubility of starch-treated OPEFB fibers composites decreased from approximately 35% to 27% as the loading percentage of fibers increased from 0 to 20%. The NaOH treatment on fibers removes non-cellulose substances, increases the crystallinity of the fibers, and thus improves the compatibility with the matrix (Nagarajan et al., 2021).

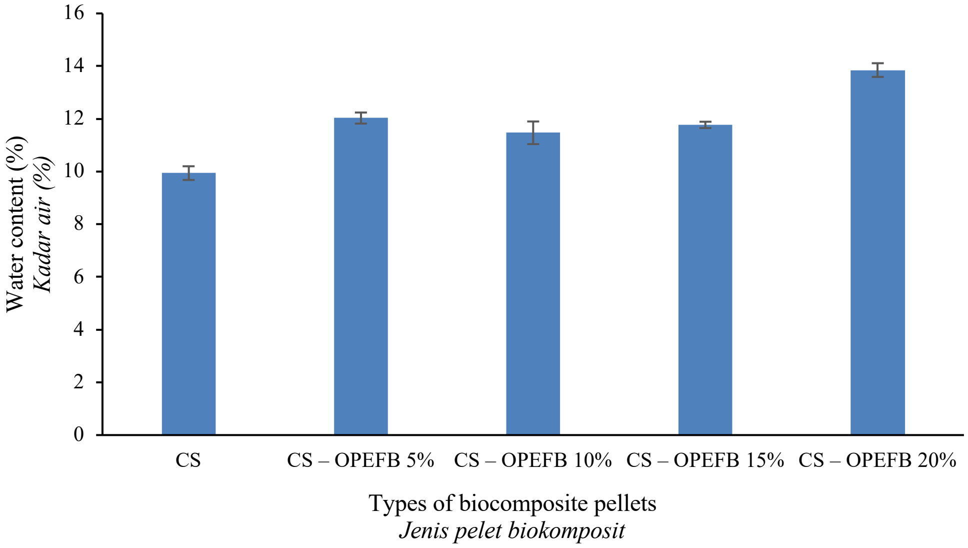

Figure 5. The water content of biocomposite pellets from the mixture of starch with varying OPEFB concentrations. CS = cassava starch and OPEFB = oil palm empty fruit bunch

Gambar 5. Kadar air pelet biokomposit dari campuran pati dengan konsentrasi TKKS bervariasi. PS = pati singkong; TKKS = tandan kosong kelapa sawit

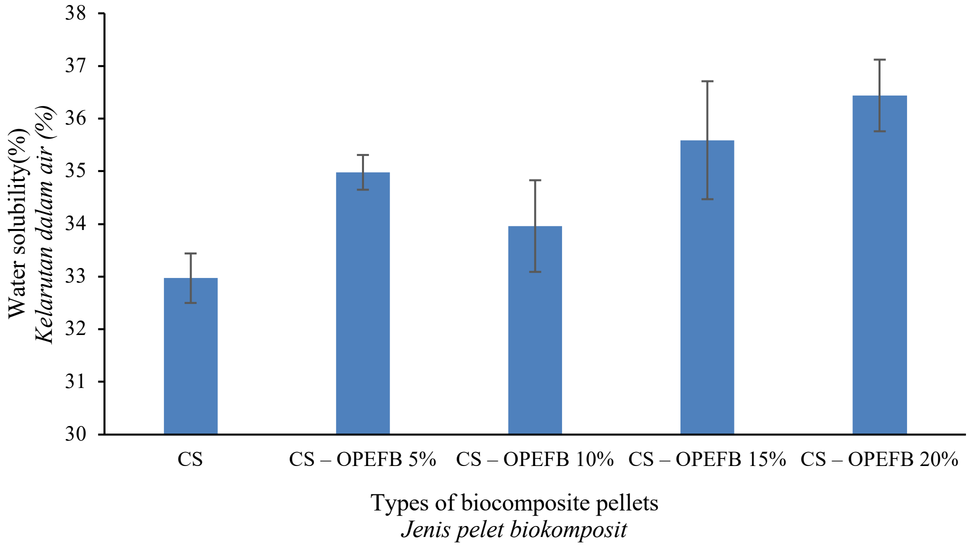

Figure 6. Water solubility of biocomposite pellets from the mixture of starch with varying OPEFB concentrations. CS = cassava starch and OPEFB = oil palm empty fruit bunch

Gambar 6. Kelarutan dalam air pelet biokomposit dari campuran pati dengan konsentrasi TKKS bervariasi. PS = pati singkong; TKKS = tandan kosong kelapa sawit

This study used raw OPEFB fibers as composite material instead of cellulose or microcrystalline cellulose as it tried to cut and shorten the fiber treatment process and the treatment cost. The biocomposite pellets produced have the potential to be applied for rigid packaging products, such as bowls and glasses. Biocomposite pellets can be used in their application for final products by mixing them with polypropylene pellets to improve their performance.

Conclusion

Biocomposite pellets were successfully fabricated from a mixture of cassava starch and OPEFB fibers up to 20% OPEFB concentration. The biocomposite pellets' characteristics tend to be darker in color (dark brownish gray) with a fibrous texture. The addition of OPEFB fibers (≥10%) in biocomposite pellets led to agglomeration structures. The density of biocomposite pellets ranges from 1.322 – 1.407 g cm^{-3}, with water content ranging from 9.94-13.85 % and solubility in water ranging from 32.97-36.44%.

Acknowledgment

This research was financially supported by PT Perkebunan Nusantara (PTPN) IV and VIII, contract number 090113/KS-RPN/IX/2022. We also thank Khairy Yunda Maharani and Muhammad Dzaki Arkaan, who helped carry out the research activities.

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