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Evaluations of the mechanical and physical properties of galangal root-poly(butylene-succinate) (PBS)-based biocomposite

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ABSTRACT

Various agricultural waste materials, such as cassava stems, pineapple leaves, banana peels, and corn pulp, were developed into natural biodegradable packaging, adding value to the agricultural waste. Hence, this research aimed to develop the biocomposites and inspect their mechanical and physical properties. Fresh galangal root waste was washed and dried at 80°C for 12 hours and then ground to achieve a particle size of 250 microns (GR250) and 400 microns (GR400). Then, they were mixed with PBS at the ratio of PBS: GR250 at 80:20 wt.%. Later, the mixtures were passed through the extruder, and the plastic strands were obtained. Later, these plastic strands were shredded into small pellets called biocomposite pellets. These pellets were formed by heat at 150°C for 5 minutes under a pressure of 10 MPa to obtain the biocomposite specimens. Then, they were assessed the mechanical properties (tensile strength, impact strength, and flexural strength). Also, the physical properties (water absorption, density, morphology, and percentage of natural degradation) were performed. The results could imply that adding GR250 and GR400 into PBS-based biocomposite could cause reductions in structural integrity and elasticity. PBS/GR biocomposites would assert less impact force. The results could reflect that PBS/GR250 and PBS/GR400 biocomposites had more ability to resist bending stresses than neat PBS. PBS/GR400 biocomposites tended to degrade faster, as supported by microstructure observation and lower density compared to PBS/GR250. It could be concluded that the galangal root waste could be added value by developing into a based-biocomposite. Galangal root waste can produce biocomposite food containers that can resist bending stresses. Biocomposite food containers have a natural biodegradable property and environmentally friendly aspects.

Keywords: Biocomposite, Value-added, Galangal root, PBS, Agricultural waste

INTRODUCTION

Plastic has outstanding properties, such as being cheap, lightweight, strong, and durable. These properties make plastic popular, and its usage continuously increases in various aspects. The production of plastic has a low production cost. In addition, modern technology can produce plastic with various properties according to needs. In terms of the environment, the increasing use of plastic has led to increased plastic waste. According to the Ministry of Natural Resources and Environment's report, there were up to 2.76 million tons of plastic waste [1]. Because plastic has properties that are difficult to decompose and deteriorate, plastic waste remains in the environment for a long time. This plastic waste causes a significant burden in management and disposal. This inevitably results in environmental problems.

Plastic could also contaminate food and be harmful to human health. Plastic creates toxic smoke in the air and carbon dioxide gas when burnt which causes global boiling. Many international organizations, such as the Food and Agriculture Organization of the United Nations, have launched a campaign to reduce the amount of food damage and ultimately reduce the amount of waste employing reduction of the overall environmental impact [2].

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Biopolymers, made from natural and environmentally friendly materials, have been developed for packaging applications, particularly in the pharmaceutical sector, aiming to sustainably reduce environmental pollution [3]. There were also many research had reported the studies of utilization of food production waste by mixing it with natural fiber. For example, the development of packaging was created from mango leaves [4]. A mixture of chitosan with olive pomace from the olive oil production process to produce packaging and film [5-6]. In addition, there was a large number of research on producing films and packaging made from various wasters, for instance, fruit peels [7] or pomegranate peels [8], potato waste with gallic acid [9], chitosan with polyphenols extracted from apple peels [10], and the use of chitosan mixed with apricot seed oil [11] or mango peel [12].

In addition, there were also research reports on the development of food additives, dietary supplements, new products such as pasta, milk, butter, crackers, cookies, etc., or the development of antioxidants in the form of powder or foam, or natural extracts developed from vegetable and fruit processing waste [13].

PBS is a biodegradable polymer made from bio-based biodegradable plastics. PBS is one of the most promising alternatives due to its good mechanical, thermal, and barrier properties, making it suitable for use in various applications. [14]. Additionally, during decomposition, PBS converts its product into water and carbon dioxide gas, releasing fewer toxic substances into the environment. For this reason, PBS is becoming another alternative plastic. Pure PBS is expanding in packaging and single-use products (disposal items) such as agricultural films, engineering materials, and medical materials [15]. However, the research on the value-adding of the galangal root waste was still limited. Research on how to increase the value of agricultural waste would be meritorious to the extent of its application.

The previous study developed corn waste from the corn milk production process into a biocomposite mixed with PBS at 80:20 (wt.%) by studying 2 levels of corn residue particle size (CK250 and CK400). The results showed that PBS/CK400 specimens showed better tensile force and had higher %elongation at break than PBS/CK250. However, PBS/CK250 had better flexural strength than PBS/CK400. The biocomposite contained a smaller size of corn residue powder and exhibited a higher density than PBS/CK400. The results of monitoring the natural degradation in soil burial tests showed that PBS/CK400 tended to have a faster degradation rate (R^2=0.8677) than PBS/CK250 due to its larger particle size [16].

Study investigated the effects of PBS and cassava pulp (CP250 and CP400 micron) with 5 different ratios (PBS100/CP0, PBS90/CP10, PBS80/CP20, PBS70/CP30, and PBS60/CP40) on the properties of biocomposites. The study found that the melt flow index of biocomposites significantly decreased with the increase in the CP content, as observed in both CP particle sizes. It was also observed that using CP400 for PBS80/CP20 resulted in a higher melt flow index than CP250. The PBS/CP biocomposites were shown to improve Young's modulus and hardness. However, tensile strength, elongation at break, and impact strength of the PBS/CP biocomposites decreased with increased CP. The addition of CP250 provided good particle dispersion and compatibility in the biocomposite matrix. This study suggested avoiding adding CP levels greater than 40% to maintain the structure's integrity. This study concluded that the biocomposite developed by PBS/CP could be useful in commodity packaging, outdoor plant pots, and injection molding as eco-friendly and biodegradable materials [17].

Nakhon Ratchasima province is an important area that produces fresh galangal supplies for the Northeastern markets [18]. Fresh galangals, both young and mature galangals, are generally used for many dishes. At the end of fresh-cut galangal production, many galangal roots are abundant on the farm. Usually, the farmer often leaves them in the agricultural field for compost, landfill, or burning.

Due to the environmental pollution resulting from plastic usage, especially single-use plastic packaging, this research attempted to add value to the abundant galangal root. Hence, this study aimed to develop a biocomposite composed of galangal root and PBS. It was hoped that this research would add value to agricultural waste. Moreover, this could result in the widespread adoption of various types of single-use food packaging that are environmentally friendly and made from renewable resources. Ultimately, this may promote the development of BCG (businesses that focus on sustainability and environmental responsibility).

MATERIALS AND METHODS

(a) Preparation of galangal root powder

The preparation of galangal root powder began with washing with tap water for 15 minutes to remove all impurities. Afterward, the galangal roots were cut into shorter pieces and dried in a hot air oven (FD115, Binder, Germany) at 80°C for 12 hrs with constant turning and mixing. At the end of drying, the final moisture of the dried galangal root was 17% (dry basis). The dried galangal root fragments were ground with a fine grinder at a 1 mm sieve size. After that, they are sieved with a stainless-steel sieve (W.S. Tyler, USA) to obtain galangal root powder with a size of 250 (GR250) and 400 (GR400) microns (Figure 1 (a), (b)). The galangal root powder was stored in a zip-lock bag and in a desiccator until used.

(b) PBS and preparation of PBS/GR biocomposite specimen

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PBS bioplastic pellet (PBS, grade: B3C03) was purchased from Teamplas R&D Co., Ltd. (Chachoengsao province, Thailand). The melting temperature and the density were about 170-200°C and 1.50 g/cm³ [17], respectively (Figure 1 (c)).

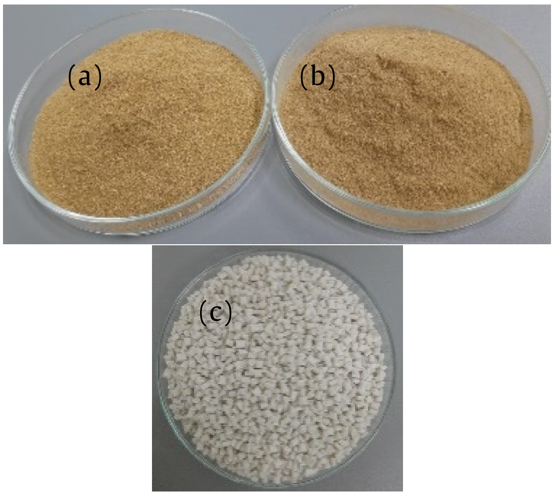

Figure 1 (a) GR250, (b) GR400, and (c) PBS pellets.

The PBS pellets were dried in a hot air oven to remove moisture before being used at 60°C for 3 hrs. After that, they were mixed with galangal root powder, which was labeled as PBS/GR250 and PBS/GR400. The mixing ratio between PBS and galangal root was 80:20 (wt.%). This ratio was chosen based on the previous study. Studies revealed that the optimal ratio between PBS and agricultural fiber was 20%. This is because lower or higher percentages of agricultural fiber than 20% reduced mechanical strength [19-21].

After that, the mixture was melted until homogeneous using an internal mixer to obtain the biocomposite in the form of strands. After the biocomposite strands obtained from the melt were allowed to cool down, they were passed through a shredder to obtain the biocomposite pellets. The biocomposite specimens were obtained by extrusion molding of biocomposite pellets with a hot extrusion machine at 150°C under a pressure of 10 MPa for 5 minutes.

Therefore, three different sizes of specimens needed to be prepared according to the standard methods for mechanical inspections. The specimens for tensile strength testing were produced according to ASTM D638 [22] in the form of type 2 dumbbells with a thickness of 3.2 mm, a narrow section width of 6 mm, a narrow section length of 33 mm, and a total length of 115 mm The specimens for impact resistance evaluations had dimensions of width x length x thickness of 13 mm x 63.5 mm x 3.2 mm by ASTM D256 [23]. The specimens for flexural strength assessments were prepared according to ASTM D790 [24] with a width x length x thickness of 12.7 mm x 127 mm x 3.2 mm. The specimens were stored in a desiccator until the tests were performed.

(c) Mechanical property evaluation

The PBS/GR250 and PBS/GR400 biocomposite specimens were tested for mechanical properties, including tensile, impact, and flexural strength. Experiments were conducted with 5 replicates. The tensile strength was performed using a universal testing machine (LLOYD, model 30k Plus, UK) with a 10 kN load cell and a 50 mm/minute crosshead speed. The specimens were held vertically in the Y-axis during the test and clamped at both ends. The tensile strength, stress, and % elongation at break were assessed. The tensile strength is the force that can be applied to a material before it yields (stretches irreparably) or breaks. The tensile stress is stress developed by a material bearing a tensile load [22].

For the Izod impact test of the specimens, a pendulum impact tester (INSTRON, model CEAST 9050, USA) was used according to ASTM D256. The specimens were clamped at one end and fractured after being hit by a pendulum with an impact energy of 2.7 Joule.

For the flexural strength test, during testing, the specimens were clamped at both ends in a flat position with a support span-to-depth ratio of 16:1. The tests were performed on a universal testing machine (LLOYD, model 30k Plus, UK), using the three-point bending fixture at a crosshead speed of 5 mm/minutes. The flexural strength (MPa), flexural stress, and Flexural Modulus (GPa) were measured.

(d) Physical property assessment

Physical property evaluations for biocomposites include water absorption, density, morphology, and weight loss percentage during burial tests. Experiments were conducted with 5 replicates.

According to ISO 535 (2014) [25], the water absorption test was conducted by weighing a 12 mm x 63 mm x 3 mm specimen before and after immersing it in distilled water for 60 minutes. The water absorption was calculated using Equation (1)

$$\text{Water absorption} = \frac{W_f - W_i}{W_i} \times 100 \qquad (1)$$

where

W_i = weight of the specimen before immersion, g

W_f = weight of the specimen after immersion, g

The density was calculated from the relationship between weight and volume. The weight of the specimen was measured directly using a three-point precision electrical balance (AND brand, model FX-300i, Japan). The volume of the specimen was determined via the dimensions of the specimen using the digital vernier caliper, and then the volume was calculated. The density of the specimen was calculated using Equation (2).

$$\rho = \frac{m}{v} \qquad (2)$$

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where ρ = density, g/cm^3
m = mass, g
v = volume, cm^3

The natural degradation of the biocomposite specimen was determined by a burial test. The burial test was conducted at the field of the Department of Agricultural Machinery Engineering, Faculty of Engineering and Technology, Rajamangala University of Technology Isan, Nakhon Ratchasima, Mueang Nakhon Ratchasima District, Nakhon Ratchasima Province, at latitude 14°59'06.9"N and longitude 102°07'11.0"E. The soil was a clay loam type. The six holes with dimensions of width 25 cm x length 15 cm x depth 15 cm [26] were constructed. All specimens were weighed and recorded as initial values to monitor the natural degradation. After that, five specimens were buried in each hole. Burial periods were 0, 7, 21, and 121 days. After excavation, the specimens were washed with tap water to remove dirt and dried in a hot air oven (FD115, Binder, Germany) at 60°C for 24 hrs. to remove moisture. The specimens were then weighed. The natural degradation was expressed as a percentage of weight loss and calculated using Equation (3).

$$\text{Weight loss, \%} = \left[\frac{(W_i - W_f)}{W_i}\right] \times 100 \qquad (3)$$

where
W_i = weight of the specimen before burial test, g
W_f = the weight of the specimen after burial test, g

The morphological investigation for the microstructure of the biocomposite was conducted on the cross-section area and the surface after the tensile test using a scanning electron microscope and energy dispersive x-ray spectrometer (SEM-EDS) (Quanta 250, FEI Company, Hillsboro, Oregon, USA) without prior specimen preparation. Morphological observations of each sample were performed at 300x and 1000x magnifications.

RESULTS AND DISCUSSION

Galangal root was the waste from the fresh-cut galangal process. This study turned this waste into fiber-reinforced biopolymer composites. Additionally, they measured the mechanical and physical properties. The results are revealed below.

(a) Mechanical properties of PBS/GR biocomposites

Table 1 displays the tensile strength, stress, and %elongation at break. PBS/GR250 presented the higher tensile strength, tensile stress, and %elongation at break at 22.19 MPa, 8.87 MPa, and 1.72%, respectively. Besides, it was observed that PBS/GR400 illustrated the lower tensile strength, tensile stress, and %elongation at break at 21.06 MPa, 7.66 MPa, and 1.16%, respectively. Furthermore, studies reported tensile strength, tensile stress, and %elongation at break of neat PBS at 32.73 MPa [20], 29.28 MPa [17], and 17.50% [20], respectively. It was seen that the tensile strength, tensile stress, and %elongation at break of both PBS/GR250 and PBS/GR400 samples were lower than pure PBS. This could reflect that adding GR250 and GR400 into PBS-based biocomposite could cause reductions in structural integrity and elasticity, similar to the previous observation [17]. The present study could suggest that pure PBS was stronger and more flexible than the PBS/GR biocomposites. The reduction of elongation of the PBS/GR biocomposites might be affected by the decrease in the deformability of the rigid interface between the fiber and the matrix [27]. However, another study found an opposite trend. This study revealed that adding 20% surface modification of jute fiber with 80% PBS greatly increased tensile strength and tensile modulus [19].

Table 1 Tensile test results.

Parameters	PBS/GR 250	PBS/GR 400	Neat PBS
Tensile strength (MPa)	22.19 ±1.19	21.06 ±1.16	32.73 [20]
Tensile stress (MPa)	8.87 ±0.99	7.66 ±1.62	29.28 [17]
Elongation at break (%)	1.72 ±0.50	1.16 ±0.23	17.50 [20]

Table 2 Impact test results.

Parameters	PBS/GR 250	PBS/GR 400	Neat PBS
Absolute energy (%)	1.73 ±0.27	1.66 ±0.32	-
Impact strength (kJ/m²)	1.55 ±0.26	1.45 ±0.28	6.20 [20]
Energy (J)	0.04 ±0.01	0.04 ±0.01	-

Generally, the impact test indicates the material's toughness and displays it via absolute energy, impact strength, and the absorbed energy during sudden loading. The impact strength of PBS/GR biocomposites was evaluated, and the results are shown in Table 2. This study observed that PBS/GR250 showed absolute energy, impact strength, and energy at 1.73%, 1.55 kJ/m^2, and 0.04 J, respectively. Meanwhile, absolute energy,

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impact strength, and energy of PBS/GR400 were at 1.66%, 1.45 kJ/m^2, and 0.04 J, respectively. The lower impact strength compared to neat PBS observed by the present study was in line with the previous report [28]. This could be due to the fiber matrix interface playing a major role between matrices; consequently, a poor matrix interface could lead to weaker impact resistance.

It was reported that the impact strength of neat PBS was at 6.20 kJ/m^2 [20]. It could be implied that adding galangal powder of 250 and 400 microns into PBS-based biocomposite decreases the toughness drastically. Pure PBS could resist more impact force due to PBS having flexibility, but PBS mixed with natural fiber would not resist the impact force [29]. It may be influenced by the arrangement of the physical bonding structure between the fiber particles of the filler material and the matrix while the two materials were melted together [30-31].

The flexural test indicates the bending property of the material under three-point loading conditions. The flexural properties of PBS/GR biocomposites were measured, and the results are displayed in (Table 3). This study observed that the flexural strength, flexural stress, and Flexural Modulus of PBS/GR250 were 14 MPa, 13.93 MPa, and 3,239.16 MPa, respectively. The flexural strength, flexural stress, and Flexural Modulus of PBS/GR400 were 13 MPa, 11.03 MPa, and 2,807.02 MPa, respectively.

Table 3 Flexural strength test results.

Parameters	PBS/GR 250	PBS/GR 400	Neat PBS
Flexural strength (MPa)	14 ±1.00	13 ±0.80	45 [21]
Flexural Stress (MPa)	13.93 ±3.06	11.30 ±2.81	-
Flexural Modulus (MPa)	3,239.16 ±398.13	2,807.02 ±506.93	1,250 [32]

Table 4 Water absorption and density test results.

Parameters	PBS/GR 250	PBS/GR 400
Water absorption	0.00	0.00
Density (g/cm³)	1.16 ±0.01	1.05 ±0.02

The flexural strength of neat PBS was reported at 45 MPa [21]. Comparing the flexural strength between PBS/GR and the neat PBS, it is seen that adding galangal powder into PBS-based biocomposite could cause a reduction of flexural strength more than 3 times. The Flexural Modulus of the pure PBS was reported at 916 MPa [21] and 1,250 MPa [32]. The present study observed a massive increase in the Flexural Modulus of PBS/GR250 and PBS/GR400, similar to the reports [19, 21]. These two studies reported that PBS biocomposites reinforced with surface-modified jute fiber, PBS-bagasse, PBS-coconut shell, PBS-curaua, and PBS-sisal increased their Flexural modulus drastically.

The large increase in the Flexural Modulus could be due to the rough surface of the galangal particle promoting the adhesion with the PBS matrix, improving the Flexural Modulus [21]. Compared to the neat PBS, the present study observed that PBS/GR biocomposites presented a higher property of the Flexural Modulus at 2.6 and 2.2 times for PBS/GR250 and PBS/GR400 biocomposites, respectively. These results could suggest that they might have more stability, longevity, and load-bearing capacity than neat PBS.

Tensile, impact and flexural tests of the PBS/GR250 biocomposites presented slightly higher than PBS/GR400 biocomposites. This could be due to the similarity of surface interference between PBS and galangal fiber.

(b) Physical properties of PBS/GR biocomposites

The water absorption of PBS/GR biocomposite was assessed, and the results are shown in Table 4. According to ISO 535, when measuring the water absorption at a specific time (60 min.), it was observed that the water absorption of PBS/GR250 and PBS/GR400 were shown at zero. These results suggest that PBS/GR did not have the water absorption characteristics after 1 hr. A study of the water absorption percentage of the PBS80/curaua20 biocomposite presented the continued increasing trends of the water absorption percentage after 8 hrs [33]. This study observed that the water absorption percentages were at 0.8 wt.% after the first hour and increased to 1.5 wt.% after 8 hrs. The study reported that the neat PBS presented the lowest water absorption of 3.25% after two days of immersion in distilled water at room temperature [30]. They observed that the water absorption of pure PBS became stable after eight days and remained at 3.53% until day 14.

The present study revealed that zero water absorption percentage after 1 hr could be affected by the water molecules having difficulty accessing the PBS/GR fiber in the matrix domains [34], which is related to the fiber distribution and properties. The crucial factor that affected the water absorption was the formation of voids during the melting process, resulting in structural inhomogeneity between copolymer and PBS [35]. However, many factors, such as porosity, lumen size, fiber-matrix adhesion [36], fiber distribution, fiber length, fiber shape, and fiber content [33], were able to influence the water absorption behavior of the biocomposites.

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The density of the PBS/GR biocomposite was also determined, and the results are shown in Table 4. It was observed that PBS/GR250 and PBS/GR400 presented densities of 1.16 and 1.05 g/cm^3. It could be noted that PBS/GR250 presented smaller particles and tended to have higher density than PBS/GR400. Generally, smaller particles have a larger total surface area than larger particles of the same material. The smaller particles could also pack more tightly due to increased surface interactions and the potential for better interparticle contact. Therefore, PBS/GR250 presented higher density compared to PBS/GR400. However, natural fiber is less dense (1.2-1.6 g/cm^3) than other materials [37].

The PBS/GR biocomposites were observed in their natural decomposition by soil. The results of weight loss are illustrated in Table 5. Figure 2 demonstrates the microstructure of both biocomposites at 0 days and 121 days.

The PBS/GR biocomposites were observed in their natural decomposition by soil burial. The results of weight loss are illustrated in Table 5. Figure 2 demonstrates the microstructure of both biocomposites at 0 days and 121 days.

Table 5 Percentage of weight loss during burial test.

Burial day	PBS/GR 250	PBS/GR 400
0	0	0
7	0	0
21	0.289 ±0.09	0.498 ±0.06
121	7.480 ±0.50	15.600 ±0.23

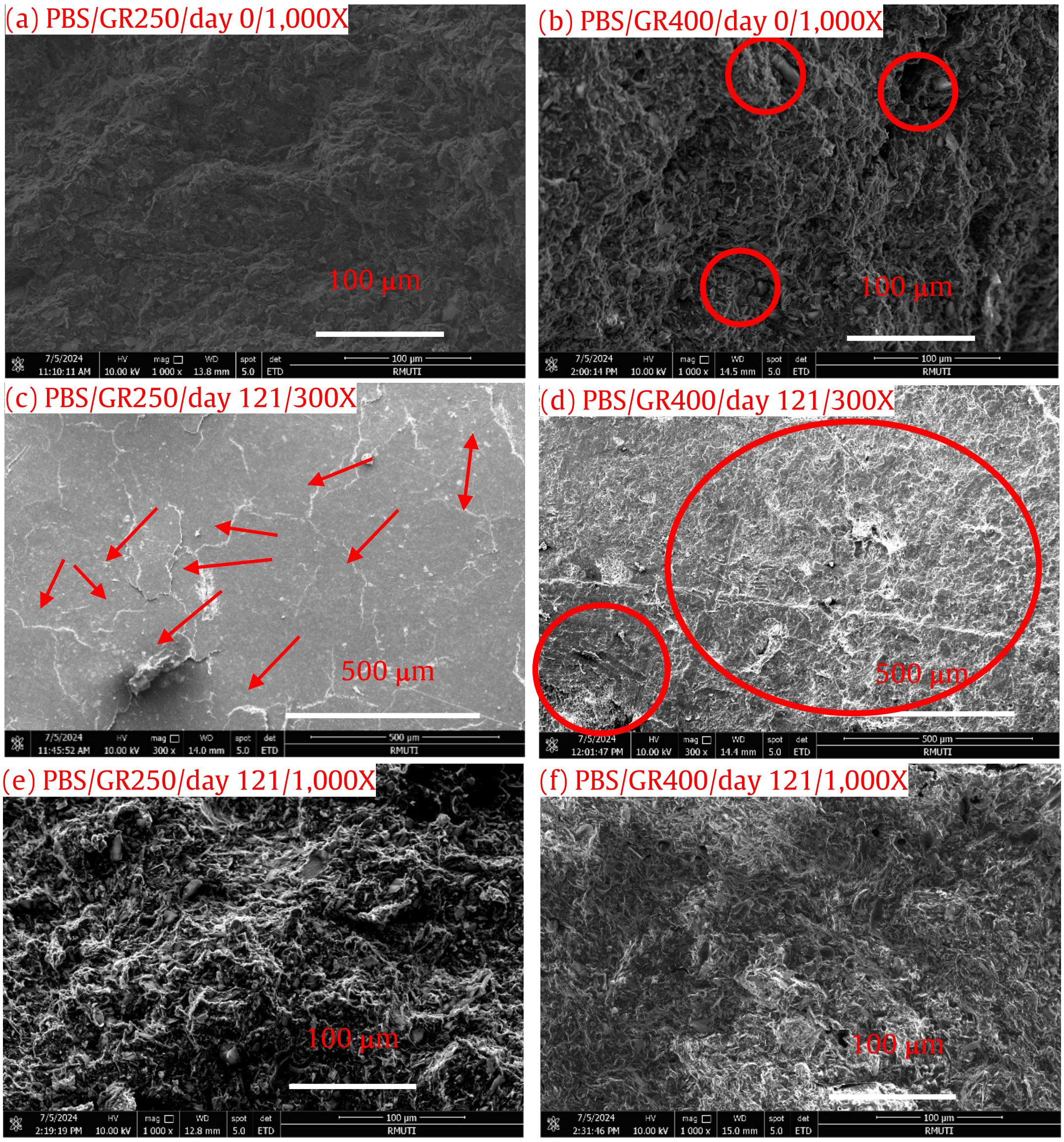

Figure 2 Morphological observations on the structure of PBS/GR biocomposites at day 0 and day 121 during burial test at 300X and 1000X magnifications (a, b, e, f display cross-section area; c, d display surface area). The red arrows in (c) point out cracks. The red circles in (d) indicate many cracks and broken surfaces.

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The results of monitoring the natural degradation by burial test were conducted on days 0, 7, 21, and 121. It was found that the percentage of weight loss of PBS/GR400 increased by double compared to PBS/GR250 after burial for 121 days. Figure 2 (b) displays the coarse particles (red circles) of galangal root powder compared to PBS/GR250 (Figure 2 (a)).

Considering the weight loss percentage of PBS/GR biocomposites, it could imply that PBS/GR400 tended to degrade well compared to PBS/GR250. This was consistent with the morphological observation in Figure 2 (d). PBS/GR400 on day 121 presented the structure as degraded and promoted a huge number of cracks (Figure 2 (d), (f)). PBS/GR250 also observed many cracks on the surface, but they were less than those observed by PBS/GR400 (Figure 2 (c)).

These could initiate the collapse of the structure as supported by a loosened structure (Figure 2 (c), (d)). The finding of lower density at 1.05 g/cm^3 of PBS/GR400 could also support the morphological observation after 121 days of burial test. Lowering density could relate to loosening surface interactions and lower interparticle contact, resulting in faster degradation in PBS/GR400.

CONCLUSIONS

This research used the leftover galangal roots from the fresh-cut process of galangal to be mixed with PBS biopolymer. The objective was to develop a biocomposite composed of galangal root and PBS. The benefits of the present study would be adding value to agricultural waste, reducing waste, and protecting the environment by further developing this waste. PBS/GR250 and PBS/GR400 biocomposites could resist the flexural forces. All biocomposite samples did not show the percentage of water absorption, and all samples present low density. PBS/GR400 presented twice higher weight loss percentages than PBS/GR250. This implied that PBS/GR400 tended to have a faster natural degradation rate. The results of this study could be further developed into single-use food containers. The production sectors for food packaging technology are already available for a wide range of industrial productions. It would be possible to do this in a mass-production manner, and it could be more cost-effective.

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