

WATER-ABSORBENT MATERIALS FROM PINEAPPLE STEM STARCH AND NATURAL FIBER AGRICULTURAL WASTES

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Abstract

This study investigates the fabrication of biodegradable water-absorbent materials using pineapple stem starch (PS) in combination with plasticizers and agricultural fiber waste. Glycerol and polyethylene glycol were applied as plasticizers to enhance flexibility and water uptake. Agricultural fibers - namely hemp core, pineapple leaf non-fibrous material (NFM), and whole grain leaf (WGL) - were blended with PS and additives (carboxymethyl cellulose and sodium alginate) and processed via twin-screw extrusion. The resultant composites exhibited water absorption capacities ranging from 100% to 400% after 5-24 hours of immersion. WGL-based materials showed superior water uptake compared to hemp and NFM. The addition of 5 wt% CMC and SA marginally improved water absorption and pellet cohesion. These biodegradable composites show promise as eco-friendly alternatives for use in domestic cat litter applications.

Keywords: Water Absorption; Pineapple Stem Starch; Hemp Core; Non-Fibrous Material; Whole Grain Leaf

Introduction

Starch, a glucose-based polymer composed of amylose and amylopectin, is a renewable and biodegradable material commonly used in the development of bioplastics. Its physicochemical properties, particularly gelatinization behavior and molecular branching, influence its potential applications. Natural fibers, such as hemp and pineapple leaves, are valued for their high strength and lignin content, making them ideal

reinforcement materials in biocomposites. In response to increasing demand for eco-friendly domestic cat litter products, this study explores the development of water-absorbent, flushable materials using pineapple stem starch combined with agricultural residues and environmentally benign additives.

Starch is a polymer of glucose, the basic units of which are anhydroglucose units linked by an α -

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glycosidic linkage at C1 of one glucose unit to C4 of the next. At the end of the starch molecule there is an anomeric carbon (C1) not bound to any other molecules. Therefore, each starch molecule has a reducing end. Starch molecules are divided into two main types based on their size and arrangement. Amylose is small and has few branches, and amylopectin is large and heavily branched. Thermoplastic starch (TPS), from pineapple stem an underutilized agricultural waste. Thailand, a major pineapple producer, generates approximately 1.65 million tons of fruit and 2.7 million tons of post-harvest waste annually Japheth *et al.* (2018). After fruit harvesting, the estimated waste from pineapple cultivation across the country is about 2.7 million tons per year. Instead of burning agricultural waste, converting it to valuable starch could reduce pollution. TPS, especially from pineapple stem waste - rich in amylose and starch content - is highly biodegradable and economically attractive but has poor mechanical properties Nakthong *et al.* (2017).

The chemical composition of natural fibers consists of the four main polymers cellulose, hemicellulose, pectin, and lignin. Leaf fibers are suitable for use as a reinforcing material in composite production because of their higher strength and toughness than fibers from other plant parts. This is especially true for flax, ramie, hemp, and pineapple leaves. The leaf part consists of a group of fibers connected by lignin. This research focuses on using dried and ground hemp core and pineapple leaves as structural reinforcing agents, which, when processed into micrometer-sized particles, strengthen the water-absorbing materials.

The domestic cat has become ubiquitous worldwide. Materials necessary for keeping cats indoors, such as cat litter that can be disposed of by flushing it down the toilet or burying it in the ground, are single-use materials in high demand Dittrich *et al.* (2024). Bentonite clay-based cat litter is widely used due to its excellent absorbent and clumping properties. Many clumping cat litters derived from bentonite contain crystalline silica, a substance classified as a human carcinogen. Inhalation of silica dust is associated with severe health outcomes, including silicosis and an increased risk of lung cancer, in humans. Chronic exposure to silica particles may also pose respiratory health risks to companion animals, including cats, potentially resulting in pulmonary dysfunction and long-term complications Chen *et al.* (2025). To address this issue, researchers have investigated water-absorbing materials prepared from eco-friendly material like starches, such as cassava starch or corn starch, mixed with plasticizers and additives containing hydroxyl

groups. These water-absorbing materials naturally decompose. The starch investigated in this study is derived from the pineapple stem. It contains a different amylose content compared to starches from other plants and is combined with pineapple leaf pulp or other natural fibers that act as structural reinforcing agents. By studying the effect of plasticizers such as glycerol and additives like sodium carboxymethyl cellulose (CMC) and sodium alginate (SA), composites have been developed that can effectively absorb water Bhatia *et al.* (2024). The addition of plasticizers enhances the flexibility of the composites, thereby optimizing physical properties, reducing costs, and maximizing the utilization of agricultural waste.

The aim of this work is to develop a biodegradable cat litter made from pineapple stem starch as the primary material. In this work, the amounts of plasticizer, hemp core, pineapple leaf non-fibrous material (NFM), and pineapple whole grain leaf (WGL) were studied in the preparation of water absorbent materials by twin screw extrusion. The plasticizer used was glycerol, and the additives to enhance the aggregation were CMC and SA. Water absorption capacity, clumping efficiency, and water solubility were investigated, and the crystal structures of the water-absorbent materials were studied by X-ray diffraction.

Materials and Methods

Materials

Hemp stalk was milled and sieved to a particle size less than 180 micron (80 mesh), NFM with particle size less than 180 micron (80 mesh). Pineapple whole grain leaf (WGL) was prepared from fresh pineapple leaf, which was cut into about 5mm length, same method as Kengkhetkit *et al.* (2014). WGL is composed of fibrous and non-fibrous materials. The former is designated PALF and the latter NFM. PALF and NFM were separated from WGL by sieving with steel wire mesh number 60. WGL was used without sieving (Figure 1). Pineapple stem starch was prepared from the dried pineapple stem. Dry pineapple stem, obtained from TCP HERB, Thailand. The pineapple stems were ground and sieved through 80 mesh screen, and the fibrous material was filtered out. The pineapple stem starch (PS) was used without further purification.

Glycerol from KemAus and polyethylene glycol (MW400) from Sigma Aldrich were used as plasticizers. CMC and sodium alginate of food grade were used as additives to improve the water absorption and clumping efficiency. Benzoic acid from KemAus was used for preservation.

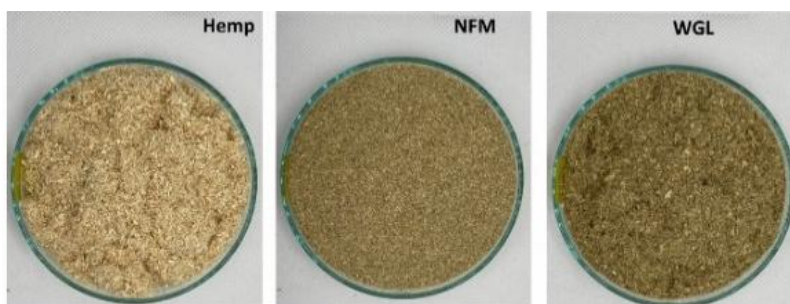


Figure 1. Agricultural wastes used in this work

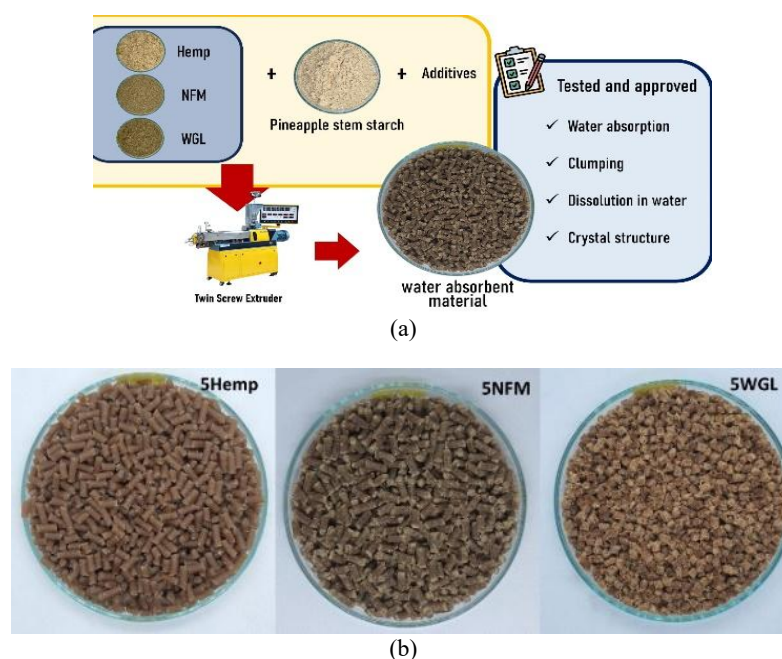


Figure 2. a) Diagram of water absorbent material preparation b) Absorbent materials from twin screw extruder

Sample preparation

Water absorbent materials were prepared by twin screw extrusion. The study compositions are shown in Table 1. Samples were prepared with different compositions while maintaining a constant 55 wt% of pineapple stem starch. Apart from hemp, NFM, and WGL, the remainder of the composition consisted of glycerol. Benzoic acid solution (0.05 wt%) was added to all samples as a preservative. The hemp core, NFM, and WGL were baked at 70°C for 24 h and mixed thoroughly with PS, glycerol, and the corresponding additives. The mixture was left for 24 h before being added to the twin screw extruder. The compounding process was conducted using a co-rotating twin - screw extruder (LABTECH model LTE16-40). The temperature profile during extrusion was set at 90°, 100°, 110°, 120°, 130°, 140°C from the feed zone

to the metering zone. The screw speed was maintained at 100 rpm throughout the extrusion process. The extruded composite material, with a diameter of approximately 1 mm, it is cooled by air and cut into pellets, as shown in Figure 2(a) and Figure 2(b). Samples with polyethylene glycol as a plasticizer could not be prepared by twin screw extrusion, so the results in this work will focus on glycerol as a plasticizer.

X-ray diffraction (XRD)

The crystal structures of pineapple stem starch powder (1-2 g) and the water-absorbent materials were observed using X-ray diffraction (XRD) (Cu K α , 1.54 Å, Philips Model X'Pert Pro). Diffraction patterns were obtained at room temperature in the 2 θ range between 5° and 40° with a 0.01° step size.

Table 1. Proportions of pineapple stem starch, plasticizer, and additives used in the preparation of absorbent materials

Samples	Hemp (wt%)	NFM (wt%)	WGL (wt%)	Additives	
				CMC (wt%)	SA (wt%)
5H	5	-	-	-	-
5H:5CMC	5	-	-	5	-
5H:5SA	5	-	-	-	5
10H	10	-	-	-	-
5NFM	-	5	-	-	-
5NFM:5CMC	-	5	-	5	-
5NFM:5SA	-	5	-	-	5
10NFM	-	10	-	-	-
10NFM:5CMC	-	10	-	5	-
10NFM:5SA	-	10	-	-	5
5WGL	-	-	5	-	-
10WGL	-	-	10	-	-

Water absorption

Samples (approx. 1 g) in a net bag were immersed in distilled water (500 mL) in a plastic cup. Each experiment was carried out in triplicate. The samples were soaked in water for 1 to 5 minutes and for 24 hours. Samples were removed from the water every minute for the first 5 minutes and dried on tissue paper for 5 minutes before weighing. The percentage of water absorption was calculated according to Formula 1.

$$\text{Water absorption \%} = (W_2 - W_1) \times 100 / W_1 \quad (1)$$

where W_2 is the weight of absorbent material after immersion and W_1 is the initial weight before immersion.

Clumping test

Distilled water (10 mL) was injected into absorbent material samples (100 g) and left for 5 minutes. A cat litter scoop was used to separate the wet and dry pellets. The dry pellets were taken out and weighed then recorded as “% weight residue,” representing the material remaining after wetting. Experiments were repeated in triplicate. This method was modified from the clumping percentage test from a previous study by Saikeaw *et al.* (2021).

Solubility test

Samples of pellets (approximately 10 g) in a net bag were immersed in distilled water (1,000 mL) and left for 24 hours prior to examining their physical appearance. In another method, samples were dissolved in water, and photos were taken before and after standing overnight to monitor any precipitation of the material, compared with those of commercial cat litters.

Results and Discussion

X-ray diffraction (XRD)

The pineapple stem starch exhibited robust peaks at 2θ values of 15.0° , 17.1° , 18.0° , and 23.0° , along with fainter peaks around 10.0° , 11.3° , 20.1° , 26.6° , 30.2° , and 34.0° , indicative of the A-type crystalline arrangement (Figure 3) Tessanan *et al.* (2023). These sharp peaks disappeared after gelatinization of pineapple stem starch under heat and shear force. This confirms that thermoplastic starch films exhibit an amorphous structure. The pineapple starch of crystalline form A was converted into V-type upon formation of thermoplastic starch. This is because the starch granules are destroyed by heat and shear forces Nakthong *et al.* (2017); Yeaprayoon *et al.* (2023), and due to the addition of a plasticizer that penetrates the starch granules and breaks the hydrogen bonds that hold the starch molecules together.

Figures 4a to 4c show that the percentage of water absorption of the absorbent materials is in the range 100-250% over the first 5 minutes. The water absorption values after 1 min of immersion of commercial cat litters A, B, and C were 286.74 ± 3.31 , 167.64 ± 14.44 , and 212.71 ± 17.01 , respectively. The results reveal little difference between the hemp and NFM. However, WGL exhibited higher water absorption than hemp core and NFM. This is due to a high amount of cellulose in WGL (70.98%) compared with hemp (45.66%) and NFM (43.69%) Kengkhetkit *et al.* (2014). CMC and SA are used as thickening agents in the food industry and are readily soluble in water. In this

work, higher amounts (>5 wt%) of CMC and SA were also explored, but due to their high viscosities, the samples could not be extruded by twin screw extrusion. However, only 5 wt% of additive may be insufficient to improve the water absorption.

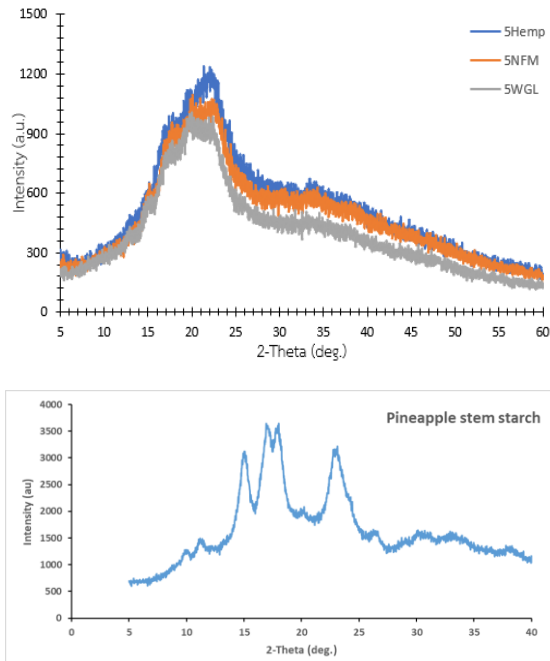


Figure 3. XRD patterns of absorbent materials prepared from pineapple stem starch and various agricultural waste materials

In the case of the hemp absorbent material, the water absorption increased slightly when CMC and SA were added. This may be due to the high lignin content in hemp (10.2%) compared with NFM and WGL (4-7%) Kengkhetkit *et al.* (2014). In addition, 2.5wt% urea solution was also examined, resulting in a slightly lower percentage of water absorption than of distilled water (100% for urea solution and 130% for distilled water after 5 minutes of immersion). After 24 hours of immersion in distilled water, the percentage of water absorption increased by 300-400% for some samples (Figure 5). It appears that an increasing amount of natural agricultural waste can improve water absorption. The use of 15% NFM resulted in a 270% increase in water absorption (data not shown in Figure 5). The composite material, consisting of 15 wt% hemp and 15 wt% WGL, could not be extruded from the die due to the die size limitation (1 mm), resulting in a high torque of the extrusion machine.

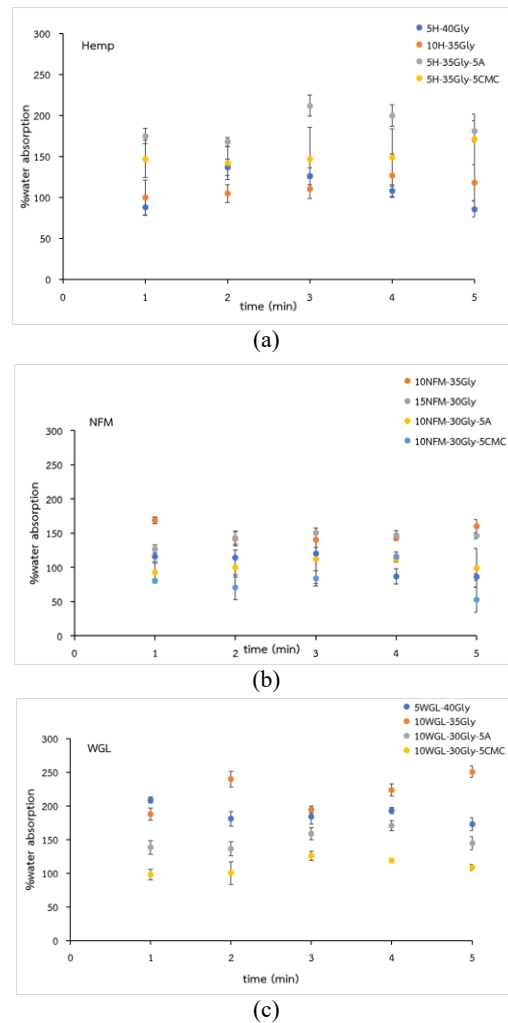


Figure 4. Percentage water absorption of composites prepared with various agricultural wastes for 1-5 mins: (a) hemp core, (b) NFM, (c) WGL

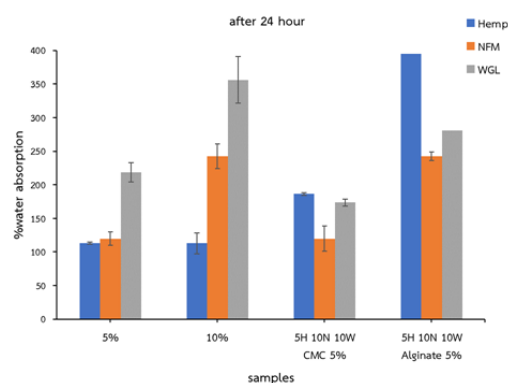


Figure 5. Water absorption of absorbent materials using various agricultural wastes after 24 h of immersion

Clumping efficiency

For these composite materials to serve as effective cat litter, the clumping efficiency is an important property, as illustrated in Figure 6. The residue weight of the absorbent materials after the injection of distilled water is reported in Table 2. A high percentage weight residue indicates a significant amount of material left behind after use, this could be called clumping efficiency which is a desirable property for a cat litter. For some samples, the dry and wet pellets could not be separated after testing, so no data is provided in the table. Three commercial cat litters, which claimed to be biodegradable and suitable for disposal in a toilet, were also investigated. The water-absorbent materials based on pineapple stem starch were found to give slightly lower % weight residues than the commercial materials. In practice, the higher % percentage of residue, the less amount of cat litter taken out, and the better the clumping efficiency. Clumping efficiency refers to the ability of agglomeration with litter particles that come into contact with liquid, resulting in the adhesion force of clumping agents and other additives that adhere to the particle surface. Thus, cat litter with high clumping efficiency can be easily scooped out of dry cat litter. Additionally, the minimum clumping of cat litters required by consumers is at least 60 percent Vaughna *et al.* (2020); Saikeaw *et al.* (2021). In this study, it was found that pineapple stem starch-based material can provide a sufficient clumping capacity for the cat litters.



Figure 6. Clumping test of 5NFM sample

Table 2. % Weight residues of absorbent materials compared with commercial cat litters

Sample	% weight residue
5NFM	66.12 ± 4.37
5WGL	69.64 ± 3.84
10WGL : 5SA	72.44 ± 0.40
15NFM	74.73 ± 10.02
Commercial A	90.32 ± 0.35
Commercial B	-
Commercial C	82.53 ± 1.23

Note: Commercial A, B and C are Tofu, Water hyacinth, and Cassava starch cat litters, respectively.

- means that the wet and dry pellets could not be separated.

Solubility test

The absorbent materials were immersed and stirred in distilled water, and two commercial cat litters were also examined. All the samples could be easily dissolved in water within 5 minutes. After 24 hours, some sedimentation of solids was observed in all samples, as seen in Figure 7. After 24 h, all samples reveal similar behavior. These tests were conducted to assess the feasibility of flushing the absorption material down the toilet after use.

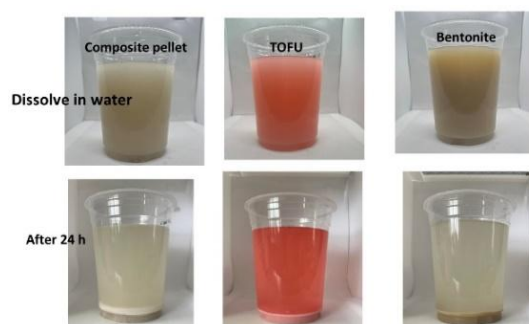


Figure 7. Solubility test in water after 24 hours compared with commercial cat litters

Conclusions

Biodegradable water-absorbent composites were successfully developed using pineapple stem starch and agricultural waste fibers through twin-screw extrusion. The incorporation of whole grain leaf and non-fibrous material significantly enhanced water uptake, while carboxymethyl cellulose and sodium alginate contributed to improving pellet cohesion. Among the materials studied, NFM-based composites exhibited the best balance between water absorption, clumping efficiency, and processability. These results underscore the potential of utilizing agricultural by-products to develop sustainable, environmentally friendly alternatives for cat litter and related absorbent applications. Some characteristics are still in process, such as the analysis of odor absorption and antibacterial.

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