Morphological analysis of chemically umbut palm stem starch granules using scanning electron microscope

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Abstract. The purpose of this research was to determine the morphological structure of modified umbut palm stem starch. The method used was an experimental method with a Completely Randomized Design (CRD). The treatments in this study were A (modified with 1% CH_3COOH), B (modified with 2% HCl), and C (modified with 4% H2SO4). The result of this research is the morphological structure of umbut palm stem starch undergoes changes in granular structure after hydrolysis with various acids. Particle size of the sample with 100x magnification in each treatment are treatment A 177.76µm, treatment B 130.86 µm and treatment C 85.25 µm. Based on the results of morphological analysis of modified umbut palm stem starch using several acid treatments that starch granules in treatment B have more changes in granule shape, as can be seen from the destruction of irregular starch shapes and the presence of many flakes has a shape like a split stone and a rather smooth surface. Implications of the results of the research are known that the umbut palm stem starch that has been modified using different acid treatment is seen morphologically undergoing changes in structure or shape.

1 Introduction

Oil palm trunks consist of vascular bundles and parenchyma tissue that have the function of storing food reserves in the form of carbohydrates, most of which can be in the form of sugar and starch. The concentration of starch in the core part of the oil palm trunk is known to be higher than that in the outer part and will increase with increasing height [1]. Umbut palm stem is the base of the young palm fronds found about 30 cm from the fresh fruit bunches of oil palms. Umbut palm stem can be obtained by replanting oil palm plants that are no longer productive. The utilization of umbut palm stem is very necessary to be developed because it can be used as an alternative source of carbohydrates [2].

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Starch is composed of polysaccharides from α-D-glucose molecules, and its molecules consist of linear amylose and branched amylopectin fractions. Amylose and amylopectin molecules have a role in the physical and chemical properties of starch, so that they affect the level of solubility and swelling of starch granules [3]. Starch is mostly stored in tubers, stems, fruits, and seeds. Starch granules have the main components of amylose (15%–30%) and amylopectin (70%–85%) and contain other materials such as fat (5%–10%). Starch granules have a size of 1μm - 100 μm. Structures such as shape, size, size distribution, and crystallinity of granules vary depending on the botanical properties of the starch source [4]. Natural starch has limited industrial applications because it is not resistant to heat and shear stress and quickly undergoes retrodragation and syneresis. Therefore, it is necessary to modify natural starch in order to improve the functional properties of starch so that its application is wider [5].

Modifications to starch can vary, one of which is chemical modification of starch using acid solutions. Hydrolysis of starch using acid can have properties such as viscosity that tends to be stable at both high and low temperatures, resistance to mechanical treatment, and thickening power that is resistant to acidic conditions and high temperatures, making this modified starch very suitable to be applied on an industrial scale [6].

To see the morphology of the modified umbut palm stem, a scanning electron microscopy test was conducted. Scanning electron microscopy (SEM) is an electron microscope that uses electron beams to obtain images of the shape of the sample surface. SEM analysis is carried out to see the morphological structure of a sample [7]. The morphological structure of the cellulose of the palm oil stems at the time before and after the chemical and mechanical treatment has different forms. At the chemistry treatment with H_2O_2 the surface of the fiber has begun to be orderly but there are still irregular fiber parts, cut into shorter fibers, but there is some part that is damaged in the crystalline structure [8]. This study has a gap only analyses the morphological structure of starch that has been modified by hydrolysis of various acid. The purpose of this research was to determine the morphological structure of modified umbut palm stem starch.

2 Methods

2.1 Materials and equipment

The main raw materials used in this study were replanting waste of umbut palm stem and then taking the stump, namely at the base of the young palm fronds, which is about 30 cm from the fresh fruit bunches of oil palm, acetic acid (CH_3COOH) pa, hydrochloric acid (HCl) pa, and sulfuric acid (H_2SO_4) pa, distilled water, 70% ethanol and chemicals used for testing.

2.2 Research methods

The research was started by making starch from umbut palm stem, then modification through acid hydrolysis and analysis of starch morphology using Scanning Electron Microscope. The treatments in this study were A, B and C.

2.3 Starch production

The production of umbut palm stem starch is carried out by separating the umbut palm stems and then separating the hard shell, the umbut palm stems is reduced in size and then reduced in size. The size of the umbut palm stems is added distilled water then squeezed and then

filtered with a filter cloth. The residue was removed and the starchy water was precipitated for 3 hour followed by drying for 24 hour at 50℃ [9].

2.4 Starch modification process

Umbut palm stem starch flour was hydrolyzed using 1% CH_3COOH, 2% HCl, and 4% H_2SO_4. The ratio of the material to the acid solution is 1:20. The starch suspension of umbut palm stem shoots was hydrolyzed with an acid solution at 40±5ºC. The starch was washed several times with distilled water to remove the remaining minerals. Residual starch that is neutral is filtered. The residue that was not filtered was washed with ethanol and stirred for ± 2 hours, and the starch residue was removed from the filtrate and dried in an oven at 40 ± 5 ℃ for ± 24 hours (moisture content < 10%). The dry modified starch is sieved and then uniformly sized by a 100 mesh sieve [10].

2.5 Analytical procedure with Scanning Electron Microscopy (SEM)

SEM (Scanning Electron Microscopy) is an electron microscope that uses an electron beam to obtain an image of the shape of a sample surface. SEM analysis was used to determine the morphological structure of the sample. The working principle of SEM test analysis is to view an image of the surface of an object or material by depicting the surface of the object or material by a high-energy reflected electron beam [7].

The starch powder samples were observed with SEM-EDS. The samples were placed on stubs and then coated with gold using a gold sputter coater samples were placed into the SEM microscope and observed up to 1000x magnification [11].

3 Results and discussion

Scanning electron microscopy (SEM) experiment was performed to determine the surface morphological structure. SEM test analysis results can provide information about the shape and changes of the material being tested. The principle of inspection by SEM is that changes in cracks, indentations, and structural changes of material tend to undergo changes in energy. These energy changes are emitted, reflected, absorbed, and converted into electronic waves, which are then captured and read on SEM images. The following morphological testing of modified umbut palm stem starch can be seen in figure 1.

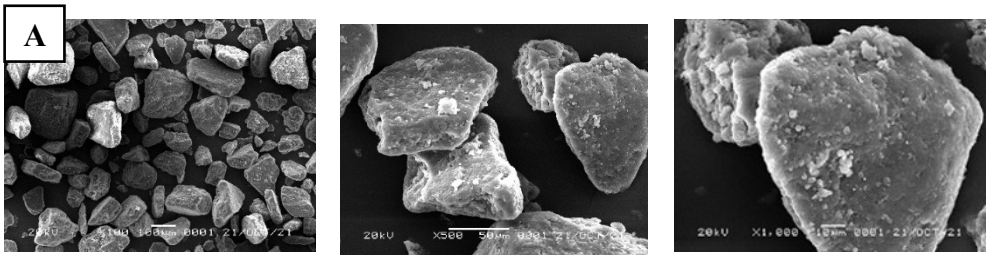

Fig. 1. Morphological analysis by SEM (Scanning Electron Microscopy) Treatment A (modified by CH_3COOH 1%).

Umbut palm stem starch in treatment A had the shape of an irregularly split stone, then with voids. In Figure 1, it is known that in starch granules that have been modified using CH3COOH the granules are damaged due to their irregular shape. It seems that some of the particle fragments are still attached to the larger particles. The shape of the starch of the umbut palm stem hydrolyzed using acid is the same as the modification of starch with a

combination of pregelatinization and steam explosion, namely experiencing irregular shape changes [1].

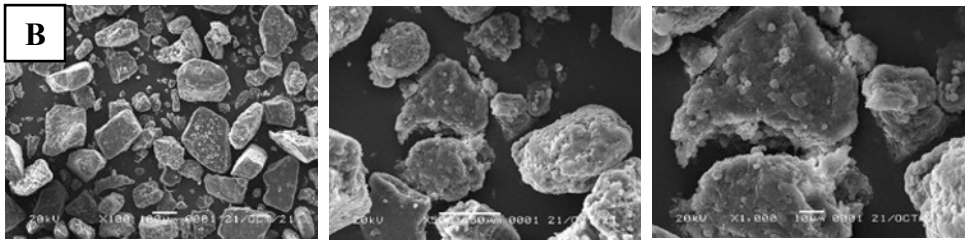

Fig. 2. Morphological analysis by SEM (Scanning Electron Microscopy) Treatment B (modified by HCl 2%).

Umbut palm stem starch in treatment B were shaped like irregularly split and scaly stones. In Figure 2, we know that starch granules denatured with HCl have damaged the structure of starch granules. It looks like the shape of starch granules is irregular and there are many flocs. The surface of the granule becomes more damaged and the granule becomes smaller due to the higher acid concentration in acid hydrolysis [1]. According to [10] it was known that the granules of starch that were hydrolysed with acid were damaged but their shape did not change. The longer the extraction time, the more the cell walls of the starch granules broke so that there was a hole in the modified starch granules.

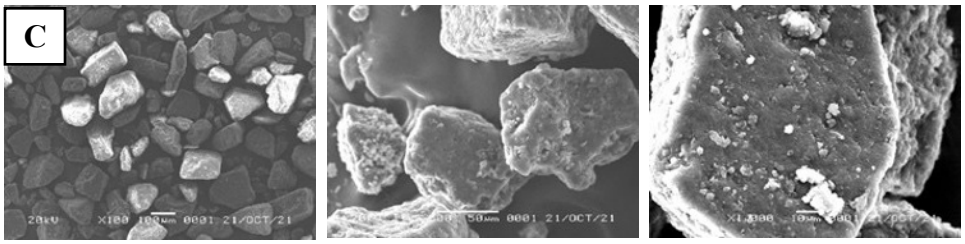

Fig. 3. Morphological analysis by SEM (Scanning Electron Microscopy) Treatment C (modified by H_2SO_4 4%).

Umbut palm stem starch in treatment C had a shape like a split stone and a rather smooth surface. In Figure 3 it is known that the starch granules that have been modified using H2SO4 have a smooth surface when viewed at 100x magnification and there are no voids in the starch granules. In the morphological analysis using SEM, measurements were made of the average particle size area in each sample as follows (see Table 1):

Table 1. The average particle size area in each sample.

No	Treatment	Average particle area (µm)
1	A: (CH_3COOH 1%)	177.76
2	B: (HCl 2%)	130.86
3	C: (H_2SO_4 4%)	85.25

Based on the results of the analysis of the average particle size of the samples with 100x magnification for each treatment, the result obtained was that the consecutive average particle size was treatment A 177.76 µm, treatment B 130.86 µm and treatment C 85.25 µm. The

difference in grain shape and size is due to the difference in content between amylose and amylopectin [12]. Furthermore starch granules can be attacked by acid hydrolysis which occurs in two stages, namely the amorphous part attacks quickly and the crystalline part attacks the amylopectin fraction slowly [12]. Acid hydrolysis easily attacks the amorphous region and the fixed crystalline region, so it does not change the particle shape much [13].

4 Conclusion

Based on the results of morphological analysis of modified umbut palm stem starch using several acid treatments including A (CH3COOH 1%), B (HCl 2%) and C (H2SO4 4%) is known that starch granules in treatment B with the use of HCl 2%, more changes in particle shape can be seen from the destruction of irregular and scaly starch shapes. Implications of the results of the research are known that the umbut palm stem starch that has been modified using different acid treatment is seen morphologically undergoing changes in structure or shape.

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