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EFFECT OF NATURAL FIBERS REINFORCEMENT ON TENSILE STRENGTH OF ACRYLIC DENTURE BASE: IN VITRO STUDY, SEM AND X-RAY DIFFRACTION ANALYSIS

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ABSTRACT

The use of acrylic (HCAR) dentures for a long time often causes problems, namely, they are easily damaged or broken. This damage occurs when used due to user negligence. One of the causative factors is the poor nature of acrylic resin (HCAR), especially in terms of impact strength. The method to increase the strength of the acrylic plate is by adding fiber. There are 2 types of fibers known in Dentistry, namely synthetic and natural fibers. Synthetic fibers are relatively more expensive, so the use of natural fibers can be an alternative, for example, ramie and banana stem fibers. This study aims to determine the characteristics of the HCAR denture base reinforced by ramie and banana stem fibers in the form of tensile strength, SEM, and X-ray diffraction and to see a comparison between the control group and the ramie and banana stem fibers groups. Research method in the form of an Experimental Laboratory, a total of 30 samples. The sample is an acrylic dumbbell shape of 75×10×3 mm in accordance with the ISO 527-1 (2019) for the tensile strength test. The concentration of fibers added was 1.6% of the sample weight. The sample consisted of 3 groups, namely control, the addition of ramie fiber, and banana stem fiber. Tensile strength testing used the Universal Testing Machine, followed by SEM and X-ray diffraction. Data were analyzed using the Oneway ANOVA test and the Post Hoc LSD test ($p < 0.05$). Results: The ramie fiber addition group had the highest tensile strength (70.06 MPa), while the banana stem fiber addition group had the lowest tensile strength (64.98 MPa). There was a significant difference in the control group and the addition of ramie fiber, and ramie fiber and banana stem fiber ($p < 0.05$). In SEM examination, it can be seen that the fracture edges and rough surfaces and cracks are different for each group and X-ray diffraction analysis shows the same results in all groups. Conclusion: The addition of ramie fiber can increase the tensile strength of the HCAR denture base, but it is different from the addition of banana stem fiber. This is in accordance with the SEM image obtained on ramie fiber, which shows a rough surface and cracks that spread across the fracture surface with solid, converging protrusions and visible cavities formed due to the fibers being pulled out of the matrix.

Keywords: *Ramie fiber, Banana stem fiber, Tensile Strength, SEM, X-ray diffraction, Acrylic denture*

INTRODUCTION

Heat-cured acrylic resin is a dental material based on acrylic resin and its polymerization uses cooking or heating. The use of dentures with a base made of heat-cured acrylic resin (Polymethyl Methacrylate) is currently still an option, although many other materials have become superior along with technological developments. This is because the heat-cured acrylic resin has a color that is close to the color of oral tissue so it has good aesthetics, is easy to manufacture, easy to clean, relatively inexpensive, and easy to polish so that its surface smoothness can last for a long time (1,2). However, dentures with acrylic resin material have weaknesses, namely, they are easily damaged and break easily after a long time of use. Damage to acrylic dentures often occurs when used in the mouth or outside the mouth due to user negligence. One of the factors that cause damage is the lack of physical properties of acrylic resin, especially in terms of impact strength, flexural forces, and fatigue during use (2,3). In addition to its physical properties, mechanical properties are also very basic aspects to determine the durability of all polymeric materials. The mechanical properties commonly measured to determine the character of a polymer are yield strength, tensile strength, elongation at break, and hardness (4). Dentures made from acrylic, with several of these weaknesses, are often damaged or broken over time, for example, due to falling when cleaning and hitting a hard floor or for other reasons.

Various methods can be used to prevent the acrylic denture base from breaking, including using reinforcement materials such as fiber. Fibers are pieces of components that form a complete elongated network, grouped into two namely natural and synthetic fibers. The price of synthetic fiber is more expensive so a new material is needed as an alternative, namely natural fiber. The natural fiber is a promising biomaterial because of its biocompatibility and Young's modulus and higher tensile strength than denture base materials. The use of Natural Fiber in the field of Dentistry and Dental technic is still rare (5–7). Water hyacinth fiber, sisal fiber *(Agave Sisalana),* banana stem fiber, and ramie fiber are some of the natural organic fiber materials that have many uses. Ramie fiber *(Boehmeria Nivea)* is produced from natural fibers derived from very hard and shiny bark. This plant has a small diameter (10-60 μm) and a white color so it meets the aesthetic requirements of a denture base (6). While the midrib is part of the banana stem. This banana plant grows a lot in the territory of Indonesia. This is because the tropical climate in Indonesia makes it suitable for the growth of banana plants. Banana plants have specific characteristics that are easy to distinguish from other types of plants. On the stem there is a midrib that has fiber, so far the fiber in banana tree trunks is just trash that hasn't been used much (7,8).

There are several studies have been carried out to strengthen acrylic resin as a denture base by adding fibers in the form of glass fiber, carbon fiber, polyethylene fiber, aramid, and metal wire (3,9). Likewise, the addition of natural fibers, namely sisal fiber was carried out by Eko Hadianto (2013), while other research in the field of engineering with the same material (resin) was carried out by Kunarto et al (2018) on banana stem fiber and water hyacinth as reinforcement composites with variations in fiber direction to tensile and bending tests (7,10).

From the description above, the author is interested in researching the characteristics of a heat-cured acrylic resin base with the addition of ramie fiber and banana stem fiber and its effect as a reinforcement for acrylic denture base, so that in the end it is expected to be useful for dental technicians and dentists in providing adding strength (reinforcement) to denture bases made of acrylic and at the same time being able to develop the use of natural fibers for fiber reinforcement materials at a fairly economical cost.

RESEARCH METHODS

This research method is in the form of a laboratory experiment with a post-test-only design with a control group design. The total number of specimens was 30 acrylic plates, divided into 3 groups, namely: 10 control specimens each (without fiber), added ramie fiber, and banana stem fiber. The natural fibers used are ramie and banana stems. For ramie fiber *(Boehmeria Nivea)* it is taken from the bark which is very hard and shiny white in color. The banana stem fiber used is taken from the stem of the kapok banana *(Musa Paradisiaca L)* which is taken inside and dried until the water content is reduced and the physical appearance of the banana stem has dried out so that the fiber is visible. Drying was carried out for 12 days by keeping the sample from becoming moldy because it would reduce the quality of the fiber.

The use of these natural fibers previously required an alkalization process with 5% NaOH solution which aims to increase the roughness of the fiber surface layer, adhesion between the polymer matrix and the fiber, and reduce water absorption in the fiber (11). The volume of fiber in this study was 1.6% of the weight of the acrylic plate with the formula: fiber volume (%) = fiber weight (gr)/sample weight (gr) x 100% (10). The specimen is in the form of a dumbbell-shaped acrylic plate from a metal master mould, with dimensions according to ISO 527-1 of 2019 75x 10x3 mm for the tensile strength test (12). Figure 1 shows a diagram of how research works in general. The specimens were made of HCAR material *(BasiQ20, Vertex, Nederlands)* and the fibers used were cut according to the length and width of the specimen plate shape, then weighed so that the fiber weight criteria were met, then the fibers were dipped in monomer until all were wetted. Polymer and monomer with a ratio of 2.4 g: 1 ml are stirred in a mixing jar. The dough is then inserted into the mold space up to a height of 1/3 of the part before the dough reaches the dough phase, the fiber is impregnated and placed in the middle 1/3 of the mold. After the dough phase is added to 2/3 of the dough and the cuvette is closed, plastic cellophane is previously layered on top of the dough and the cuvette is pressed with a hand press. The cuvette was reopened, the remaining acrylic was cleaned, then the cuvette was closed again and pressed with a table press using a pressure of 2200 psi (50 kg/cm2). The next stage is curing, namely placing the cuvette in boiling water for 20 minutes (100°C), then removing the cuvette and cooling it (according to the manufacturer's instructions). After that, the specimen was removed from the cuvette and the specimen surface was smoothed with abrasive paper numbers 360, 600, and 1000, then the specimen was cleaned with water spray. Soaking in aquabides and storing in an incubator at 37°C for 24 hours was carried out after the specimen preparation process was complete. After drying, each specimen at both ends is numbered and a mark is made in the form of a line in the middle of the specimen, then it is ready for testing and data analysis.

The Universal Testing Machine *(Shimadzu, Japan)* was used for testing Tensile strength, with a load of 10 kN, a grip distance of 50 mm, and a crosshead speed of 5mm/minute. The test method is that the specimen is placed vertically/perpendicular and the two ends are clamped and then pulled until they break (figure 2). Specimens before and after testing with UTM are shown in Figure 3. The tensile strength value is calculated by the formula ISO 527-1 (2019) (12) : ʃ = Fmax/Ao; ʃ = Tensile strength (MPa); F = pulling force (N); A = Surface area (mm2). Then the specimen with the highest value from each group was examined for its microscopic picture using a Scanning Electron Microscope (SEM) and X-ray diffraction. Analysis of the morphology of the fracture surface from the results of the tensile strength test was then carried out by a Scanning Electron Microscope (SEM) test with 200 times magnification to determine the surface morphology of fractured specimens without and with the addition of fiber. XRD test (X-Ray Diffraction test analysis) to see if there is a change in the diffraction characteristics of the acrylic plate material with the addition of fiber. In this test, specimens from 3 groups (1 specimen each) were made into powder/powder weighing 10 mg, namely by grinding with a Fraser bur and then filtering it with a filter (0.1 mm). Tests were carried out using the continuous method, the device used was XRD Empyrean with a measuring angle of 20-80°, to analyze the phases contained.

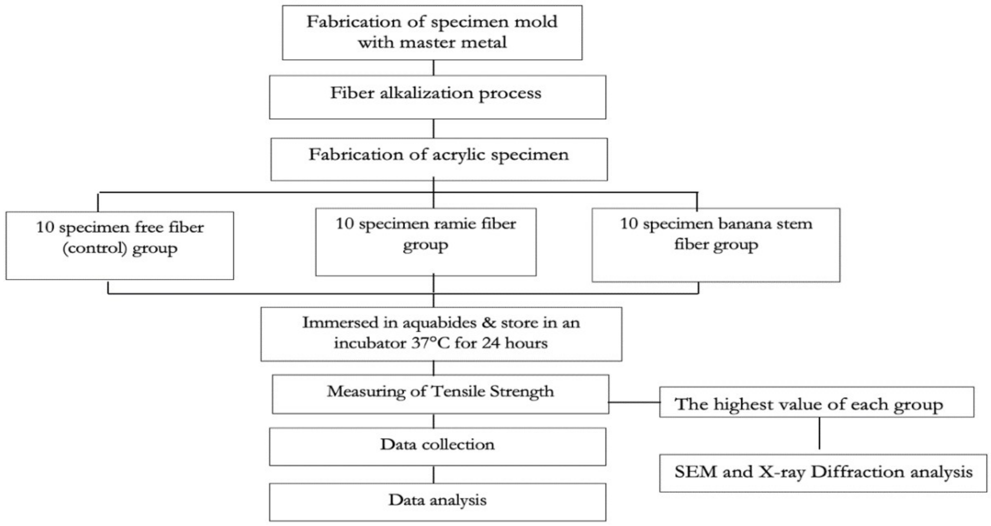

Figure 1. Research workflow diagram

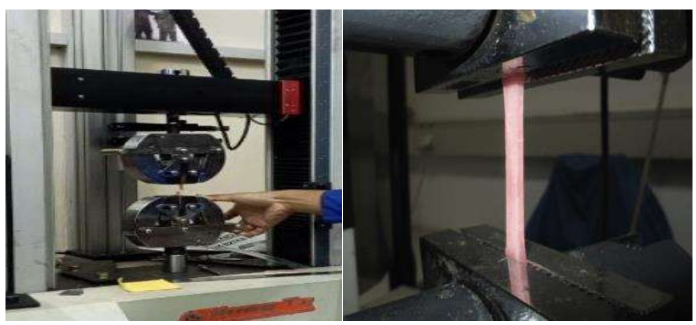

Figure 2. (A) Universal Testing Machine; (B) Specimen when pulled

Figure 3. (A) Specimen before testing (B) Specimen after testing

Data were analyzed using SPSS and after data collection, normality, and homogeneity tests were carried out and the average calculation of each group for the tensile strength test was carried out. To find out the significance, an analysis was carried out using the One way ANOVA test with a significance value of $p < 0.05$, and a follow-up test was carried out with the Post Hoc LSD test

RESULTS AND DISCUSSION

The data generated from this study were previously tested for normality using the Shapiro-Wilk test and the results obtained were normal data distribution because all groups had a p-value > 0.05, then continued with the Lavene homogeneity test to obtain data in all homogeneous groups, because the p-value > 0,05.

Table 1: Mean Tensile Strength values of HCAR plates in the free-fiber group (control), the addition of ramie fiber, and banana stem fiber (N=30).

Groups	N	Tensile strength minimum	Tensile strength maximum	Mean Tensile strength MPa ± SD
Free-fiber (Control)	10	63.98	69.03	66.40 ±1.471
Ramie fiber	10	66.86	72.80	70.06± 1.937
Banana stem fiber	10	62.91	67.68	64.98 ± 1.298

From table 1, it can be seen that the mean tensile strength value of the ramie fiber addition group has the highest mean value of 70.06 ± 1.937 MPa, followed by the control group's mean value (66.40 ± 1.471MPa) and the smallest mean value in the addition of banana stem fiber (64.98 ± 1.298 MPa).

Table 2: LSD Test - One Way ANOVA, Significance between groups of HCAR plates free-fiber (control), with the addition of ramie fiber and banana stem fiber in the Tensile Strength Test (N = 30).

Groups	Mean difference		*p*
Free-fiber (control)	Ramie fiber	-3.661	0.000*
	Banana stem fiber	1.420	0.056
Ramie fiber	Free-fiber (control)	3.661	0.000*
	Banan stem fiber	5.081	0.000*
Banana stem fiber	Free-fiber (control)	-1.420	0.056
	Ramie fiber	-5.081	0.000*

Description: *Significant

To find out the significance of the difference in the mean value of the tensile strength (MPa) test in the free-fiber group (control), the group with the addition of ramie fiber and the addition of banana stem fiber was carried out a one-way ANOVA test with a significance value of p <0.05, and because the p obtained was 0.000 (Ho was rejected) so that a follow-up test was carried out with the LSD (Least Significant Different) Post Hoc test to determine the difference in significance between the groups. It can be seen from table 3, that the significance (p) between each group (control, addition of ramie fiber and banana stem fiber) in the Tensile Strength test found a significant difference (p <0.05), namely p = 0.000 except between the

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ramie fiber group and there was no significant difference in the banana stem fiber because p = 0.056 (p > 0.05) (table 2).

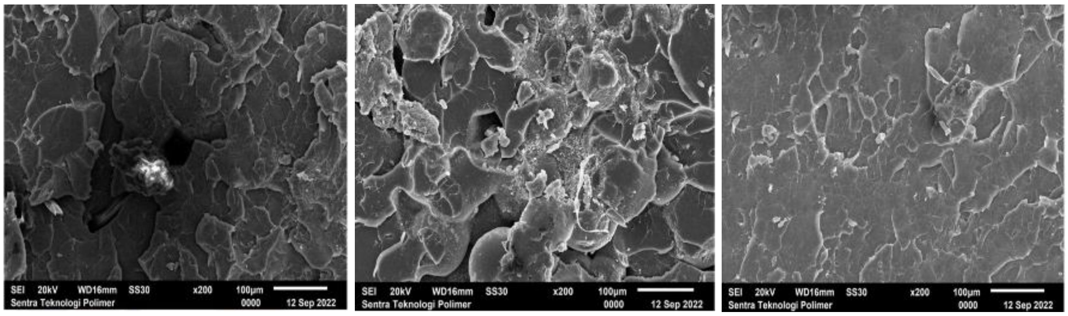


Figure 1. SEM Image of Free- fiber group

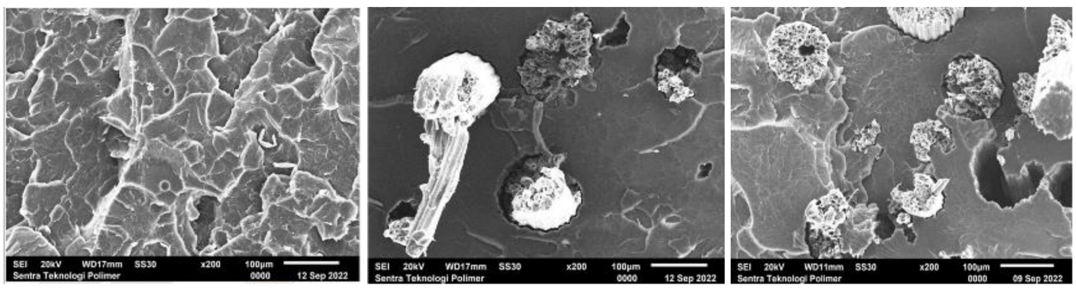


Figure 2. SEM Image of Ramie-fiber group

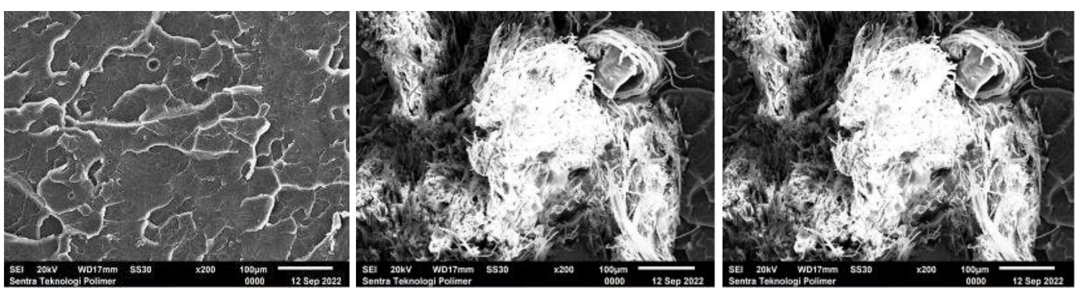
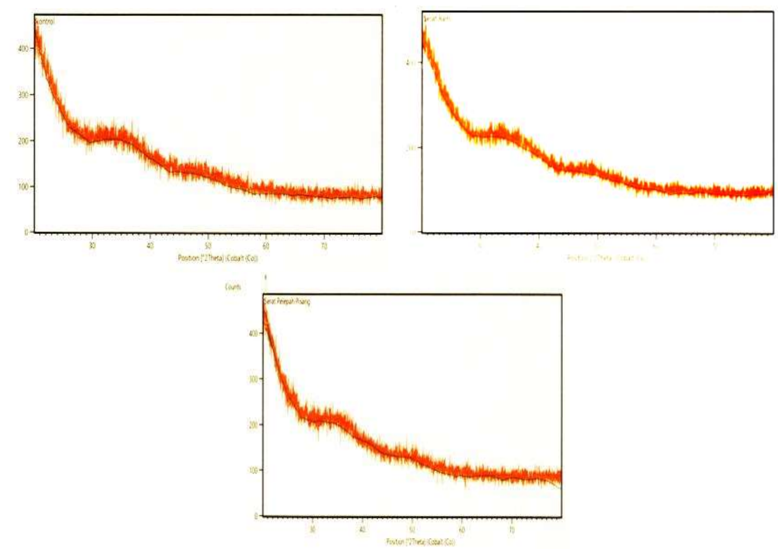


Figure 3. SEM Image of Banana stem- fibers group

Figure 1: Microscopic view (SEM) of group A from the fractured end of the acrylic plate specimen showing a rough surface and cracks spreading throughout the fracture surface. Figure 2: The SEM view of group B which shows a rough surface and cracks that are smaller in size than group A, ramie fibers can be seen protruding/sticking out of the resin matrix, but the fibers still stick together to form thick and dense clumps and you can see a cavity formed due to the fibers being stretched pulled out of the resin matrix. Figure 3: The SEM view of group C, which shows a rough surface and cracks that are almost the same size as group B and the banana stem fibers protruding/sticking out of the resin matrix but visible fibers that are more spread out to form thin and fine fibers.



(A) (B) (C)

Figure 4. Graph of X-Ray Diffraction Pattern with a measurement angle of 20 -80°
A. Control group; B. Ramie group; C. Banana stem group

Graph in Figure 4, it can be seen, the diffractogram (diffraction pattern) of the crystallinity test results with X-ray diffraction analysis showed the same results in all groups, both without and with the addition of ramie and banana stem fibers. The heat-cured acrylic resin in this study has an amorphous structure that is characterized by the absence of sharp peaks in the resulting diffractogram so that in this study the degree of crystallinity and the phases contained cannot be known.

In this study, the mean HCAR tensile strength value in the group without added fiber was 66.1167 MPa, this value was greater than previous research conducted by Rahmadita (2018), namely 50.867 MPa and Sitorus (2017), namely 53.010 MPa (13,14). However, the mean tensile strength value in this study, both groups without fiber and with the addition of ramie fiber and banana stem fiber, was still within the recommended limits according to ISO 527 specifications, which ranged from 36 -77 MPa and was greater than the tensile strength value based on ASTM D 638. i.e. 55 MPa (14). Tensile strength on HCAR dentures can be influenced by several factors, including the presence of residual monomer and invisible porosity, wasting of acrylic material during pressing so that the concentration of fibers in the specimen is not evenly distributed, and polishing manually with abrasive paper so that the roughness on the surface of the specimen is not evenly (13).

The difference in the tensile strength values of the free-fiber group between this study and the studies of Sitorus (2017) and Rahmadita (2018) may be due to differences in the HCAR denture base material used and the size of the specimen (13,14). In this study, the HCAR material used was *BasiQ20, Vertex, Nederland* with specimens in the form of flat dumbbells according to ISO 527-1 2019, namely 75x10x3 mm (21). While in research conducted by Rahmadita (2018), the HCAR material used was *GC, America* with a size of 60x12x3.9 mm, while sitorus (2019) used *GC, America* with a size of 80x10x4 mm. This difference in acrylic resin brands allows for differences in the ratio of polymer and HCAR monomers, polymerization processes, water absorption, and internal porosity that are not visible (15,16).

The use of acrylic-based ramie fiber in this study gave higher scores compared to the group free-fiber, possibly due to load transfer between the fibers and the polymer matrix and the adhesion between the two. When under load, the HCAR polymer interatomic bonds have the lowest Young's modulus. Cracks in the denture base are caused by long-term deformation because the young modulus of the HCAR denture base exceeds the stress point threshold. The added fibers increase the HCAR voltage point threshold. This is because the fibers absorb some of the load that the denture base receives, which increases the tensile strength and makes the denture base less likely to fracture (17).

In addition, in this study, the orientation of the fiber direction is the same, as well as the length and width of the acrylic plate. The orientation and direction of the fibers on the acrylic plate affect the flexural strength. The grain direction is placed perpendicular to the direction of the force, at which point the force is distributed evenly across the fiber section. This situation is in accordance with the FRC Efficiency theory (Krenchel Factor), which explains that if the fiber position is in the same direction (unidirectional) to the tensile strength, then the value is 1 and if the fiber position is perpendicular to the tensile strength, the value is 0 (18). Another study was also conducted by Lokantara (2007) regarding the analysis of orientation and processing of filter fibers, stating that 0º fiber orientation has a strong bond because the direction of the fiber is mostly in the same direction as the load strength (19). Another possibility that can increase the tensile strength value in this study, besides ramie fiber having a fairly high cellulose content, is also due to the alkalization treatment with 5% NaOH. The purpose of alkalization using NaOH is to reduce the water content in the fiber, increase the adhesion between the matrix and the ramie and increase the roughness on the surface of the ramie fiber (20). This is in accordance with the research of Maryanti (2011) concerning the effect of alkalization of coco-polyester composites, noting that the addition of the percentage of NaOH to the alkalization of fibers can increase the value of tensile strength (21). Another possibility is the highest mean in the group with the addition of ramie fiber compared to the group free- fiber and the addition of banana stem fiber because the cellulose composition of ramie fiber (65%) is higher than that of banana stem fiber (52.3%), cellulose plays an important role in the strength of the fiber itself and not easily degraded chemically or mechanically so that the tensile strength of ramie fiber is the highest (915 MPa) compared to banana stem fiber (22). In addition, there is a high content of flavonoids and tannins in banana stem fiber. Flavonoids (phenolic compounds) have a molecular weight that is smaller than the molecular weight of acrylic polymers so when they come in contact with acrylic, they will cause chemical damage to the acrylic surface. Phenol in contact can diffuse into the acrylic plate and begin to damage it chemically starting with the breaking of long chains of acrylic polymers. Damage to these compounds causes roughness on the surface layer of the acrylic, which can result in cracking or crazing and the further consequence is that the acrylic plate will decrease both its hardness and strength (23,24).

The tensile strength value of the HCAR plate with the addition of ramie fiber in this study was higher than the control group and the addition of banana stem fiber. This can be seen from the microscopic image (SEM) of this group, that the fracture surface of the specimen shows the presence of ramie fibers sticking out/protruding in a form that is still united and solid, in addition to the presence of voids on the surface of the interface fracture indicates the resistance of the fibers to fracture fault. The control group showed larger cracks compared to the fiber-reinforced group whose cracks looked small. This may be due to the presence of fibers preventing crack propagation and changes in crack direction resulting in smaller cracks between fibers. This is consistent with research conducted by Mowade (2012) concerning the effect of fiber reinforcement in HCAR on impact strength and SEM (17). However, for the addition of banana stem fiber, the tensile strength value was lower than that of all groups, this was because the content of hemicellulose flavonoids and tannins was quite high compared to jute fiber. This can be seen in the microscopic image (SEM) of the banana stem fibers sticking out but looking scattered and in the form of thin fibers.

Acrylic resin is a polymer chain consisting of repeating methyl methacrylate units. Acrylic resin has an amorphous structure. This amorphous structure is a disordered molecular chain bond. Acrylic resin is formed through a free radical polymerization process that forms polymethyl methacrylate (PMMA). This is different from crystalline which is a regular molecular chain bond (1). In this study, the presence and absence of fiber addition did not cause changes in the molecular structure of acrylic resin. The results of the diffractogram (diffraction pattern) from the results of the crystallinity test using X-ray diffraction analysis showed the same results in all groups, both without and with the addition of ramie and banana stem fibers. The HCAR in this study had an amorphous structure that was characterized by the absence of sharp peaks in the resulting diffractogram so that in this study the degree of crystallinity and the phases contained in it could not be known.

CONCLUSION

The addition of ramie fiber can increase the tensile strength of the HCAR denture base because ramie fiber has a high cellulose content and low lignin, while the addition of banana stem fiber has a lower tensile strength value than the control group and the addition of ramie fiber, due to the hemicellulose, lignin, flavonoid content. and the high tannins in banana stem fiber make it easy for chemical damage to occur, starting with the breaking of long chains of acrylic polymers. In the SEM image of ramie fiber, it can be seen that the rough fracture surface is smaller and the dense fiber is sticking out and the presence of voids indicates the fiber's resistance to pulling compared to the banana stem fiber and the control group. In this study, the presence and absence of the addition of natural fibers (ramie and banana stem) did not cause changes in the molecular structure of the HCAR plate.

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