

# A Highly Active Galam Wood Bark-derived Solid Acid Magnetic Catalyst with Properties Suitable for Hydrolysis Reaction

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**Abstract.** Galam wood is a specific plant that is distributed in Kalimantan, especially in lowland areas, shallow peat forests, and swamps. After removing the bark layer, galam wood becomes a valuable building material. The galam bark (GB) is a biomass waste that could be utilized as a solid acid magnetic catalyst (M-SA). Biomass-based solid acid catalysts have gained interest due to the need for sustainable and low-cost alternatives. Nevertheless, most of them have low reusability, poor acidity, or high production cost. To create carbon material, the GB was sized ( $\pm 60$  mesh) and underwent hydrothermal treatment in the presence of sulfonic acid at 90 °C for 8, 10, and 12 h. Then, the carbon was impregnated by 10 mmol/L of iron (III) chloride hexahydrate for 5 h and calcined at 500 °C for 1 h. The solid acid magnetic catalyst (M-SA) was produced. Based on Field Emission Scanning Electron Microscope (FE-SEM) observation, the morphological structure of galam bark changes due to the delignification and carbonization processes. The X-Ray Diffraction (XRD) showed a 75% increase in crystallinity after delignification. Fourier Transform-Infrared (FT-IR) showed the presence of the  $-\text{SO}_3\text{H}$  group at a wavelength of  $1137\text{ cm}^{-1}$ . The optimum sulfonation time was obtained for 8 h with an acid content of 0.710 mmol/g. The Energy Dispersive X-Ray (EDX) Analysis measurement showed the Fe and S content of 60.21% and 4.18% w/w, respectively. The highest total reducing sugar (TRS) hydrolysis was 1.345 mg/mL from the hydrolysis of 1% M-SA catalyst at 100 °C for 1 h. The stability of M-SA showed good performance for the 4<sup>th</sup> repeated use with a decrease of only 6.5%. Solid acid magnetic catalyst from galam bark has good acid catalyst specifications and has the potential to be developed.

**Keywords:** Biomass-derived Catalyst, Galam Bark Waste, Hydrolysis Reaction, Magnetic Catalyst, Solid Acid Catalyst

## INTRODUCTION

Galam wood has a natural distribution in several regions in Indonesia, such as Sumatra,

western Java, and Kalimantan (Hapsari *et al.*, 2023). Galam is found in lowland areas, shallow peat forests, and swampy areas. Galam wood is one of the main commodities

of the community in the area along the river and the edge of the swamp (Ardhana *et al.*, 2018). The use of galam wood as piles or stakes for buildings results in galam bark, which accumulates as biomass waste. Nowadays, biomass and industrial waste have become highly interesting issues utilized as catalysts both in research and their valuable merit (Guerrero-Pérez *et al.*, 2006; Kusriani *et al.*, 2018; Rifki *et al.*, 2024). The biomass waste can serve as an exciting candidate for fulfilling the future new material needs without any concerns for the detrimental environmental problems. The generation and conversion of bioresources and biowaste streams into value-added products, e.g., foods, feeds, bioproducts, and bioenergy, has been conducted in both existing and developing industries (Ardika *et al.*, 2024; Awasthi *et al.*, 2020; Wicakso *et al.*, 2023). Biomass waste is an ideal clean and renewable energy source with characteristics of wide sources, low prices, strong reproducibility, and less pollution. The galam bark contains 47% cellulose, 27.42% hemicellulose, 18.28% lignin, 5.72% moisture content, and 1.33% ash (Haryati *et al.*, 2017). Due to its high cellulose content, galam bark has the potential to develop as a new material. The utilization of galam bark has been studied, especially galam bark fiber (Asnan & Vebrian, 2024), particle board (Christy *et al.*, 2021), extraction color (Nintasari & Purwanto, 2016), and activated carbon (Haryati *et al.*, 2017). Furthermore, the development of catalysts based on biomass waste is a good alternative as a raw material (Lokman *et al.*, 2014). This highlights the need to develop more renewable and efficient catalysts to replace traditional homogeneous catalysts like sulfuric acid.

Catalysts play an important role in chemical processes, where more than 15

million tons of sulfuric acid have been consumed as a homogeneous catalyst, which is non-renewable, has high operational costs, and is difficult to separate because in the homogeneous phase, sulfuric acid itself can be used as a catalyst (Santoso *et al.*, 2016). On the other hand, heterogeneous catalysts are easy to separate from mixtures by decantation or filtration. Moreover, they can be reused, are stable, have minimum energy use, and do not form by-products, aligning with the concept of Go Green (Nata *et al.*, 2017b). Solid acid catalysts can be used to replace liquid mineral acids in catalytic reactions such as esterification, dehydration, and hydrolysis (Liu *et al.*, 2013). Recently, work on solid acid catalysts has been the highlight of attention from researchers for hydrolysis reactions of cornstarch (Nata *et al.*, 2015; Nata *et al.*, 2017c) and biodiesel production (Correa *et al.*, 2020; Nata *et al.*, 2017a). The performance in the reaction of carbon-derived catalysts depends on the precursor as a material for carbon production and the treatment process (Tao *et al.*, 2015). Hydrothermal carbonization (HTC) is a thermochemical process that can convert wet biomass into a carbon-enriched solid. The modification of materials by HTC could be a one-step process, which consists of several reactions both in series and in parallel: hydrolysis, dehydration, decarboxylation, condensation, aromatization, and others (Merzari *et al.*, 2018). This method is cheap, mild, and environmentally friendly, as no organic solvents, catalysts, or surfactants are used (Titirici *et al.*, 2007).

To date, HTC methods have been developed and introduced to synthesize solid acid catalysts for the production of biodiesel (Correa *et al.*, 2020; Hamza *et al.*, 2021; Nata *et al.*, 2017a), levulinic acid (Tian *et al.*, 2021), and bio-oil (Liu *et al.*, 2020). Solid acid

catalysts are derived from sulfonation of metal oxides and carbon sulfonation of organic matter (Ikhsan & Nizar, 2020). The modification of solid acid catalysts with magnetic properties facilitates the separation. Therefore, the method of incorporating magnetic materials into biomass-based catalysts is very important (Krishnan *et al.*, 2022). However, there is limited research on the utilization of galam bark into valuable materials, especially as solid acid magnetic catalysts. This research focuses on the utilization of galam bark in a solid acid magnetic catalyst with surface modification through carbonization-sulfonation and wet impregnation in iron (III) chloride hexahydrate solution for magnetic formation. The characterization of the catalyst is the form of morphological structure, crystallinity, surface area, and functional groups of the catalyst. Furthermore, the catalyst was tested for banana peel starch hydrolysis. The solid acid magnetic catalyst is expected to be a potential heterogeneous catalyst as a substitute for homogeneous catalysts.

## MATERIALS AND METHODS

### Material

The raw material as a carbon source for the catalysts was galam bark; it had been collected from Bati-Bati District, South Kalimantan, Indonesia. Sulfuric acid (98%,  $\text{H}_2\text{SO}_4$ ), iron (III) chloride hexahydrate (97%,  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ), sodium hydroxide (>98%, NaOH), potassium tartrate (99.5%,  $\text{K}_2\text{C}_4\text{H}_4\text{O}_6$ ), 3,5-dinitrosalicylic acid (98%, DNS), chloride acid (37%, HCl), and phenolphthalein were used in the analytical standard and purchased from Sigma-Aldrich, St. Louis, USA. The grade chemicals were pure and did not require purification.

### Galam Bark Delignification

Galam bark was washed with tap water, dried in an oven at 105 °C for 3 hours, and then crushed into powder form and passed through  $\pm 50/60$  mesh sieves. The GB (40% v/v) was soaked in 1% of NaOH (ASTM-D-1109-56, 1978), heated at 80 °C for 2 hours under stirring at 150 rpm. After the reaction, the solid part was washed with deionized (DI) water until neutral pH. The solid part was dried in the oven at 105 °C for 7 hours, and is the material called GB-D. The GB was designed to remove lignin and produce cellulose fiber.

### Synthesis of Solid Acid Magnetic Catalyst

The treated galam bark (GB-D) was mixed with concentrated  $\text{H}_2\text{SO}_4$  at a ratio of 1:10 (g/mL) and put in a hydrothermal reactor at 90 °C (Merzari *et al.*, 2018) with time variations of 8, 10, and 12 hours to form sulfonate groups ( $-\text{SO}_3\text{H}$ ) on the galam bark structure. The sulfonated materials were called CS-8, CS-10, and CS-12. After reaction, the particulate was washed with distilled (DI) water until the filtrate was neutral. At this stage, the acid content was checked to determine the best sulfonation time. Next step, the sulfonation results were soaked in 10 mmol/L  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  (500 mL), accompanied by 150 rpm stirring for 5 hours at room temperature. The source of  $\text{Fe}^{3+}$  was chosen as iron (III) chloride hexahydrate due to its highly soluble and easily appropriate stoichiometry in the  $\text{Fe}_3\text{O}_4$  formation. After the impregnation process, the solid part of the material was separated from the liquid and then dried and calcinated for 1 hour at 500 °C. The catalyst was washed with distilled water and dried for 3 hours at 100 °C. The dried material is called M-SA.

### Hydrolysis of Banana Peel Starch over Solid Acid Magnetic Catalyst

The hydrolysis process was carried out in a three-neck flask containing M-SA (1%, 3%, and 5% w/v), added with distilled water (30 mL) and banana peel starch (3%, w/v) for 60 minutes at 100 °C. The hydrolysis results were filtered, and the reduced sugar content (TRS) was analyzed by the DNS method.

### Characterization of Material

The surface morphology of the sample was observed by Field-Emission Scanning Electron Microscopy (FE-SEM, JOEL JSM-6500F) and sputter-coated with platinum before observation. The X-ray diffraction (XRD) was investigated using a Rigaku D/MAX-BX-ray diffractometer equipped with copper K-alpha ( $\text{CuK}\alpha$ ) radiation. The operation voltage was 40 kV, and the current for the machine was 100 mA. The Autosorb<sup>-1</sup> instrument was used to evaluate the Brunauer-Emmet-Teller (BET) surface area, equipped with nitrogen adsorption-desorption capabilities, using a Quantachrome. The functional groups on the sample were investigated by using Fourier transform infrared spectrometry (FT-IR, type Alfa II, Eco-ATR) in the wavelength range of 400-4000  $\text{cm}^{-1}$ .

### Analysis

Acid capacity, or  $\text{H}^+$  ion test, was performed by adding 50 mg of the sample to 20 mL of 2 M NaCl, stirring for 5 hours, filtering, and then taking 5 mL of the solution. The solution was prepared by adding 3 drops of phenolphthalein indicator and then titrating with 0.1 M NaOH. The content of  $\text{H}^+$  ions in the sample can be known by calculating the concentration of NaOH used, as can be seen from Eq. (1).

$$C_a = \frac{C_b V_b}{V_a} \quad (1)$$

where:

$C_a$ = concentration of the acid (mol/L)

$V_a$ = volume of the acid (L)

$C_b$ = concentration of the NaOH (mol/L)

$V_b$ = volume of NaOH (L)

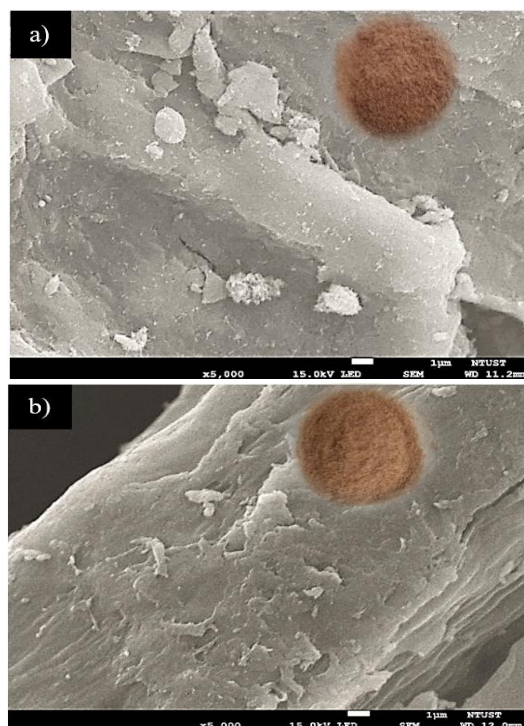
Analysis of reduced sugar content/Total Reducing Sugar (TRS) was done by adding 1 mL of DNS reagent solution to 1 mL of hydrolysis solution. The mixture was then heated in boiling water for 5 minutes and cooled for 5 minutes. The sample solution was added with 3 mL of DI water. After that, absorbance measurements were taken using a UV-Vis spectrophotometer at a wavelength of  $\lambda$  540 nm. The reduced sugar content of the sample was determined from the standard curve equation for glucose from 100 to 1000 ppm concentration. TRS measurement was done twice.

## RESULTS AND DISCUSSION

### Characterization of Galam Bark and Solid Acid Magnetic Catalyst

The complexity of galam bark should also be considered in developing an optimal pretreatment method that does not hamper the structural and compositional properties of the individual components. The primary objective in treating galam bark is to remove lignin from the lignocellulose structure, thereby facilitating easy synthesis and subsequent processes in the formation of a solid acid magnetic catalyst. Pretreatment by sodium hydroxide damages the surface of galam bark with a rough surface (Mankar *et al.*, 2021). Figure 1 presents the galam bark before and after treatment with sodium hydroxide; there is no significant change in colour after the process. Visually, the galam

bark before and after delignification is shown in Figure 1 (inset).

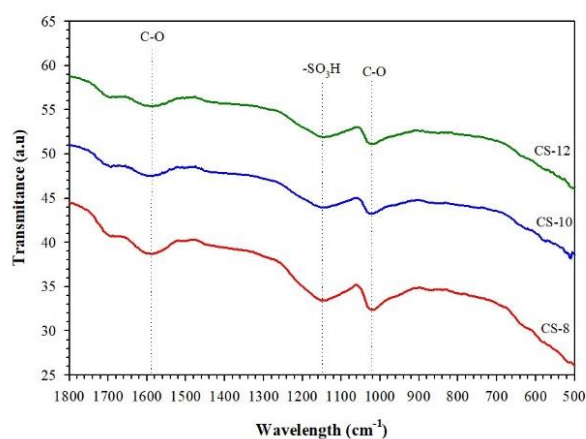


**Fig. 1:** FE-SEM images of (a) Galam bark (a) before treatment (GB) and (b) after treatment (GB-D)

The surface morphology of galam bark has a rough surface, which indicates the presence of a lignin coating. After treatment, the surface becomes rougher and looks damaged. This indicates that the treatment has been applied due to the destruction of lignin on galam bark and opening the cellulose that was previously covered by lignin (Umashankaran & Gopalakrishnan, 2021). The galam bark fiber is used as a raw material for carbonization and sulfonation in a one-step synthesis by HTC. The synthesis of carbon material involves sulfuric acid as a source of sulfonate groups, which bind to carbon during reaction (Correa *et al.*, 2020). Solid acid catalysts are expected to have a high acid concentration as active sites in the reaction. The sulfonation time is necessary to determine the optimum time of sulfonate

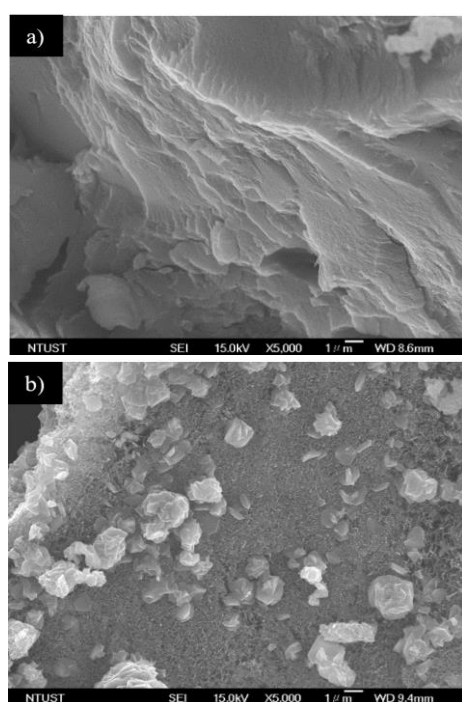
groups deposited onto the carbon surface during the process. The sulfonation will convert galam bark into carbon, where the  $H^+$  ions on the  $-SO_3H$  group are attached to the surface of carbon and act as active groups. The sulfonation time was conducted in 8 hours (CS-8), 10 hours (CS-10), and 12 hours (CS-12) and produced acid densities of 0.710 mmol/g, 0.329 mmol/g, and 0.244 mmol/g, respectively. The acid density decreased as the length of sulfonation time increased, probably due to saturation and degradation of sulfonate after 8 hours. This result is by other researchers (Fonseca *et al.*, 2020). The concentration of acid density also depends on the precursor of the carbon; the acid density of sulfonated crambe meal, bamboo, and corn cobs is about 2.19 mmol/g, 2.32 mmol/g, and 1.447 mmol/g, respectively (Nata *et al.*, 2017a; Zhu *et al.*, 2019).

The apparent sulfonate groups on carbon were confirmed by FT-IR (Figure 2). The absorption peak at a wavelength of  $1137\text{ cm}^{-1}$  shows a symmetrical  $O=S=O$  bond that indicates the  $-SO_3H$  (sulfonate) group (Zhu *et al.*, 2019), which means that the sulfonate group has been successfully formed into the galam bark carbon.



**Fig. 2:** FT-IR spectra of sulfonation catalyst prepared by different sulfonation times for 8 hours (CS-8), 10 hours (CS-10), and 12 hours (CS-12)

The order of strength of the  $\text{O}=\text{S}=\text{O}$  peak at a wavelength of  $1137\text{ cm}^{-1}$  is  $\text{CS-8} > \text{CS-10} > \text{CS-12}$ . This result proves that CS-8 has the highest acid density compared to other sulfonation time variations. The wavelength of  $1600$  to  $1020\text{ cm}^{-1}$  refers to the C-O bond (Corrêa *et al.*, 2020). The optimum time for sulfonation was achieved for 8 hours; furthermore, the CS-8 was used continuously for wet impregnation. Iron (III) tetrahydrate as a precursor for magnetic formation after calcination.



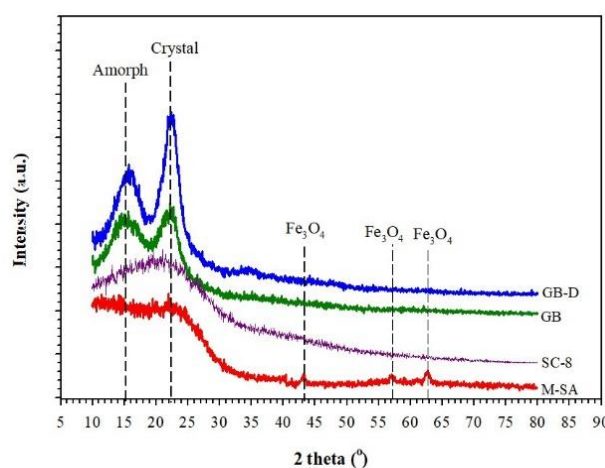
**Fig. 3:** FE-SEM images of (a) sulfonated solid acid catalyst (CS-8) and (b) solid acid magnetic catalyst (M-SA)

Figure 3 presents the morphological structure of sulfonated solid acid catalyst (SC-8) (Figure 3a) and magnetic solid acid catalyst (M-SA) (Figure 3b). The sulfonated carbon clearly shows a carbon sheet, on which the sulfonate group deposits on the surface. The surface becomes smooth compared to galam bark (Figure 1b). Furthermore, the solid acid magnetic surface with a flower-like shape was magnetic after impregnation and calcination

(Figure 3b). The EDX results on M-SA showed the Fe and S composition to be about 60.21% and 4.18%, respectively.

XRD analysis was conducted to investigate the crystal structure and determine the crystallinity index (Crl) of GB before and after delignification. The crystal and amorphous structures of the lignocellulose were investigated within the  $2\theta$  angles range of  $20^\circ$  to  $40^\circ$ , focusing on the crystal structure that exhibited a dominant peak. The amorphous structure is located at a  $2\theta$  angle between  $0^\circ$  and  $20^\circ$  with a wide diffraction pattern (Effendi *et al.*, 2019).

Based on Figure 4, the GB was identified at  $2\theta = 16.0^\circ$  and  $22-23^\circ$  for the amorphous and crystalline regions, respectively. It had a slightly higher intensity on the crystal structure. Focusing on GB-D, there was a significant increase in the intensity of the crystal structure. This could happen because the lignin layer of GB is broken due to the sodium hydroxide solution. Table 1 shows the 2 theta diffraction peaks for GB, GB-D, and the Crl number. The treatment has been effective for Crl of GB-D, which proves an increase in Crl from 19.97% up to 75%.



**Fig. 4:** X-Ray Diffraction of galam bark (GB), galam bark delignification (GB-D), sulfonated solid acid catalyst (CS-8), and solid acid magnetic catalyst (M-SA)

**Table 1.** The characterization peak of GB and GB-D and CrI

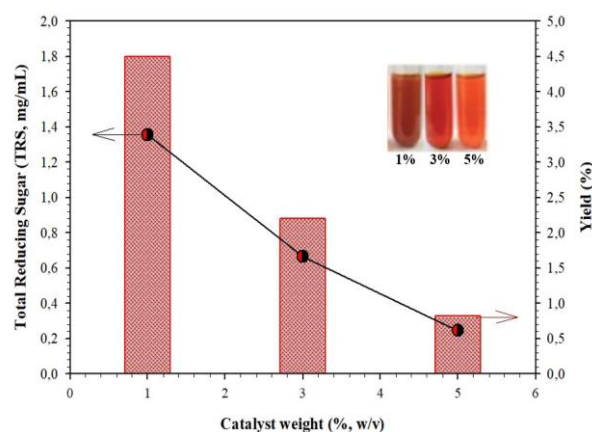
| Sample | Characteristics peak |                  | Crystalline Index (CrI, %) |
|--------|----------------------|------------------|----------------------------|
|        | Amorphous (16.0°)    | Crystals (22.5°) |                            |
| GB     | 562                  | 663              | 17.97                      |
| GB-D   | 600                  | 1050             | 75.00                      |

The diffraction of CS and M-SA differed from that of GB and GB-D; the peaks of amorphous and crystalline phases decreased due to the carbonation and calcination processes, which altered the material's structure. The diffraction broad peak formed at 2 theta 10-30°, which indicated the representative of carbon structure (Sonibare *et al.*, 2010). The peaks at 43°, 57°, and 63° characterize the magnetite (Fe<sub>3</sub>O<sub>4</sub>) particles in the catalyst within the 20° to 80° range. These findings are by the conventional shape of crystalline magnetite as recorded by the Joint Committee on Powder Diffraction (JCPDS card 39-0664) (Nata *et al.*, 2024).

### Banana Peel Starch Hydrolysis over Solid Acid Magnetic Catalyst

The performance of the prepared solid acid magnetic catalyst was tested for the hydrolysis reaction. Banana peel starch is a potential carbohydrate source for the reaction. The main constituents of starch are amylose and amylopectin. Amylose is composed of glucose units that are interrelated through 1-4 glucoside bonds, while amylopectin is a polysaccharide composed of 1-4 $\alpha$ -glycosides and has a 1-6 $\alpha$  glucoside branch chain. For starch hydrolysis, H<sup>+</sup> ions from water will attack starch at the 1-4  $\alpha$ -glucoside bond into shorter chains. The addition of a catalyst is needed to accelerate the hydrolysis reaction and act as a cutter on the 1-4  $\alpha$ -glucoside bond (Nata *et al.*, 2021). The performance of the catalyst on the

hydrolysis reaction can be seen in Figure 5.

**Fig. 5:** Solid acid magnetic catalyst performance on banana peel starch.

Operating conditions: 3% w/v of banana peel starch, volume 30 mL, 100 °C, 1 hour.

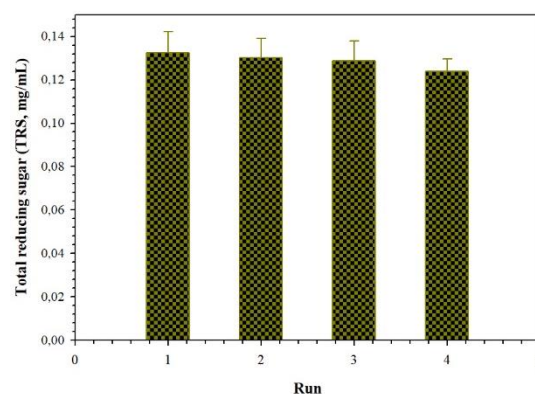
Figure 5 shows the performance of M-SA with different concentration variations of 1%, 3%, and 5% w/v in the hydrolysis reaction that produced Total Reducing Sugar (TRS) obtained at 1.345 mg/mL, 0.652 mg/mL, and 0.234 mg/mL, respectively. The yield was 4.48%, 2.17%, and 0.78% for 1%, 3%, and 5% of M-SA, respectively. The addition of M-SA as a catalyst in banana peel starch hydrolysis resulted in a 2.0-fold increase in product compared to hydrolysis without a catalyst. It means that the catalytic performance of M-SA has risen due to improved surface functionalization. The TRS produced was indicated by color intensity, which was evaluated using the DNS assay. The color of the solution changed to orange, and higher color intensity indicated a larger concentration of TRS identified (Figure 5, inset). On the other hand, by increasing the concentration of the M-SA catalyst, the TRS was reduced. This is because the high acid concentration in the hydrolysis that embeds in the catalyst causes cellulose and hemicellulose to be more easily degraded into glucose and its derivative compounds,

but along with the high concentration, glucose and other sugar compounds will be more degraded to form hydroxymethylfurfural and furfural, which causes the glucose produced to be lower (Nata *et al.*, 2021). The use of a large amount of catalyst causes the degradation of glucose to furfural, which is an inhibitor and reduces the glucose concentration. In this case, the optimal condition for the hydrolysis of banana peel starch is 1% catalyst. The performance of sulfuric acid as a homogeneous catalyst was studied in a hydrolysis reaction. The  $H^+$  ion concentration of sulfuric acid used was the same as the concentration of  $H^+$  ions in M-SA. The TRS values using sulfuric acid as a catalyst of  $1.28 \text{ mg mL}^{-1}$ . Hydrolysis reaction using M-SA has a higher TRS value of around 1.06-fold than using sulfuric acid. This is probably due to the presence of the  $-SO_3H$  group on M-SA. On the other hand, sulfuric acid only has  $H^+$  ions in a homogeneous phase.

The reusability of the catalyst was found to be lower than that achieved during the first reaction. TRS decreased by just 6.5% after the fourth repeated use, and the TRS result remained relatively steady (Figure 6). Due to the loss of an active catalyst site after washing, the acidity of M-SA was reduced by approximately 6%. When exposed to extended reaction or regeneration, the carbon support (particularly carbon derived from biomass) can partially oxidize or fragment. In addition, high-mass or derived starch molecules (amylopectin) can become physically adsorbed within mesopores or macropores of the catalyst (Mo *et al.*, 2008).

Furthermore, a solid acid magnetic catalyst is easy to handle, with nearly 98% recovery. This demonstrates that the M-SA can effectively hydrolyze banana peel starch into glucose. This result further reveals that

the prepared M-SA not only provides non-toxic ingredients, a green procedure, and easy handling, but also has good catalytic performance for the hydrolysis reaction.



**Fig. 6:** Catalytic performance of M-SA during fourth repeated use in banana peel starch hydrolysis. Operating conditions: 3% w/v of banana peel starch, 1% w/v of M-SA, volume 30 mL, 100 °C, 1 hour

## CONCLUSIONS

The high acid content, rich in sulfonate and carboxyl groups on carbon, prepared from gamal bark, could be generated by sulfonation through hydrothermal treatment. The impregnation of Fe facilitates magnetic formation on carbon, which makes it easy to separate. The magnetic acid catalyst showed good performance and stability in banana peel starch hydrolysis after the fourth use. Simple operation, high catalytic activity, and the use of waste materials are critical for the development of solid-acid magnetic catalysts. A larger scope on sulfonation temperature, quantities of sulfonic acids, and impregnation should be conducted to investigate the possibility of improving acidity and magnetic characteristics of the catalysts. The synthesized catalyst has significant potential for development in green processes and various other catalyst applications.

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