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# Use of Biopolymer Substrates as Precursors for Development of Heterogeneous Catalysts for Biodiesel Production

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ABSTRACT

The increasing demand for sustainable energy and green chemistry approaches has necessitated the development of eco-friendly catalysts from renewable resources. This study investigates the utilization of biopolymer substrates, specifically starch derived from rice (*Oryza sativa*) and corn (*Zea mays*) agricultural waste, as precursors for synthesizing heterogeneous catalysts for biodiesel production. The catalysts were prepared via wet impregnation with Iron(II) sulphate solution (0.23 g/mL) followed by calcination at 800°C for 1 hour. Comprehensive characterization was performed using proximate analysis, Brunauer-Emmett-Teller (BET) surface area analysis, Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDX), and Fourier Transform Infrared Spectroscopy (FTIR). BET analysis revealed surface areas ranging from 70.895 to 270.326 m²/g, with rice starch-based catalysts exhibiting superior performance. SEM-EDX confirmed successful iron incorporation (12.45-20.84%) and revealed diverse morphological structures including polyhedral, granular, and dendritic formations. The proximate analysis indicated moisture content between 8.70-10.95%, with carbohydrate content ranging from 61.88-82.51%. Results demonstrate that biopolymer-based catalysts possess comparable properties to conventional heterogeneous catalysts, offering an economically viable and environmentally sustainable alternative for biodiesel production.

## 1. INTRODUCTION

The global energy crisis and environmental concerns associated with fossil fuel consumption have accelerated research into renewable energy sources. Biodiesel, a biodegradable and non-toxic alternative fuel derived from vegetable oils, animal fats, or waste cooking oils, has emerged as a promising solution (Meher et al., 2006; Sarin et al., 2007). The production of biodiesel typically involves transesterification reactions catalyzed by either homogeneous or heterogeneous catalysts. While homogeneous catalysts (such as sodium hydroxide and potassium hydroxide) offer high catalytic activity, they present significant drawbacks including difficult product separation, catalyst recovery challenges, and excessive wastewater generation (Li & Xie, 2006; Arzamendi et al., 2008). Heterogeneous catalysts have gained prominence due to their ease of separation, reusability, and reduced environmental impact (Dorado et al., 2003; Tiwari et al., 2007).

Heterogeneous catalysts for biodiesel production include metal oxides, zeolites, supported metals, and alkaline earth metal compounds. Despite their effectiveness, these catalysts often require expensive raw materials, complex synthesis procedures, and energy-intensive production processes (Lee & Wilson, 2015; Singh et al., 2016). The development of cost-effective, environmentally benign catalysts from renewable resources represents a critical research priority in green chemistry and sustainable technology. Biopolymers, particularly polysaccharides such as starch, offer unique advantages as catalyst precursors. Starch, composed primarily of amylose and amylopectin, provides a carbon-rich matrix with hydroxyl functional groups that facilitate metal ion coordination (Pérez & Bertoft, 2010).

Agricultural waste streams including rice husks and corn residues represent abundant, underutilized resources that can be converted into value-added products through catalytic applications (Alabi et al., 2018; Khalid et al., 2016). The utilization of biopolymer substrates for catalyst development addresses multiple sustainability objectives: waste valorization, resource efficiency, circular economy principles, and reduced environmental footprint. Furthermore, the inherent porosity and high surface area potential of calcined starch-based materials make them suitable candidates for heterogeneous catalysis applications through the objectives to Synthesize heterogeneous catalysts from rice and corn starch waste via wet impregnation and calcination. Characterize the morphological, chemical, and physical properties of the resulting catalysts, Evaluate the suitability of biopolymer-derived catalysts for biodiesel production applications, and assess the environmental and economic advantages of this green chemistry approach.

## 2. MATERIALS AND METHOD

### 2.1 Materials

### 2.1.1 Raw materials

1. Rice (*Oryza sativa*) processing waste from local rice mills
2. Corn (*Zea mays*) residues from local agricultural processing areas
3. Iron (II) sulphate ($FeSO_4$) analytical grade
4. Distilled water
5. n-Hexane (60-80°C boiling range)
6. Petroleum ether

### 2.1.2 Equipment and apparatus

1. Electric blender and food processor

2. Stainless steel sieves (various mesh sizes)
3. Magnetic stirrer with heating plate
4. Muffle furnace (Carbolite, maximum temperature 1200°C)
5. Oven dryer (Memmert, temperature range 50-250°C)
6. Desiccator with silica gel
7. Analytical balance (0.0001 g precision)
8. Soxhlet extraction apparatus
9. Micromeritics TriStar 3000 V4.02 for BET analysis
10. Hitachi SU 3500 Scanning Electron Microscope with EDX detector
11. Airtight storage containers

## 2.2 Methodology

### 2.2.1 Starch extraction

**Corn starch extraction (Wet Method):** Dried corn grains were thoroughly washed with tap water to remove surface impurities. The cleaned grains were soaked in distilled water for 48 hours at ambient temperature to facilitate softening. The soaked corn was blended using an electric blender until a homogeneous slurry formed. The resulting mixture was filtered through cheesecloth to separate fiber residues from the starch suspension. The starch-water suspension was allowed to settle for 24 hours, after which the supernatant was decanted. The starch sediment was collected and dried under direct sunlight for 48 hours. The dried starch was pulverized using a dry mill blender and stored in airtight containers at room temperature.

**Rice starch extraction:** Rice processing waste (broken rice grains and husks) obtained from local mills was washed thoroughly with tap water to remove dust and foreign materials. The cleaned material was soaked in distilled water for 10 minutes. Subsequently, the soaked rice waste was blended using an electric blender at high speed for 5 minutes. The starch was separated from fibrous material using a fine-mesh sieve (100 mesh). The extracted starch suspension was spread in thin layers on clean trays and dried under direct sunlight for 48 hours. The dried starch was ground using a dry mill blender and stored in airtight containers.

### 2.2.2 Catalyst preparation via wet impregnation

**Impregnating solution preparation:** A dilute solution of Iron (II) sulphate was prepared by dissolving 56 g of analytical grade $FeSO_4$ in 200 mL of distilled water under continuous stirring at room temperature, yielding a concentration of 0.23 g/mL.

**Sample preparation:** Three catalyst formulations were prepared:

1. **Sample A (Mixed):** Rice starch and corn starch at 1:2 mass ratio (100 g rice starch + 200 g corn starch)
2. **Sample B (Corn):** 300 g pure corn starch
3. **Sample C (Rice):** 300 g pure rice starch

For each sample, the starch powder was transferred to a 500 mL beaker, and 100 mL of Iron (II) sulphate solution was added gradually while stirring continuously using a magnetic stirrer at 300 rpm for 10 minutes until a thick, homogeneous slurry formed. The impregnated samples were labeled and processed separately to prevent cross-contamination.

### 2.2.3 Catalyst activation

The impregnated starch samples were transferred to porcelain crucibles and placed in an oven dryer at 105°C for 1 hour to remove excess moisture. Following oven drying, the samples were subjected to calcination in a muffle furnace at 800°C for 1 hour with a heating rate of 10°C/min. This thermal treatment activates the catalyst by decomposing organic matter, creating porous structures, and facilitating iron oxide formation. After calcination, samples were cooled to room temperature in a desiccator containing silica gel to prevent moisture absorption. The final catalyst products were stored in airtight containers and labeled as follows:

1. Samples M and N: Mixed rice-corn starch catalyst (duplicates)
2. Samples O and P: Corn starch catalyst (duplicates)
3. Samples Q and R: Rice starch catalyst (duplicates)

## 2.3 Characterization Techniques

### 2.3.1 Proximate analysis

**Moisture content determination:** Clean, dry crucibles were weighed ($W_0$). Approximately 1.0 g of each catalyst sample was transferred into the crucibles and weighed accurately ($W_1$). The crucibles containing samples were placed in an oven at 103-105°C for 2 hours. After drying, samples were cooled in a desiccator and reweighed ($W_2$). The drying process was repeated until constant weight was achieved. Moisture content was calculated using equation 1:

$$\%\ \text{Moisture content} = \frac{\text{Weight loss} \times 100}{\text{Weigh (g) of sample}} \tag{1}$$

**Ash content determination:** Approximately 2.0 g of finely ground sample was weighed into clean, pre-weighed crucibles ($W_1$). Samples were initially ignited over a low flame with the lid removed to char organic matter gradually. The crucibles were then transferred to a muffle furnace and heated at 600°C for 6 hours until complete ashing occurred. Ashed samples were cooled in a desiccator and weighed immediately ($W_2$). Ash content was calculated using equation 2

$$\%\ \text{Ash content} = \frac{\text{Weight loss} \times 100}{\text{Weigh (g)of sample}} \tag{2}$$

**Crude protein determination:** The Micro-Kjeldahl method was employed following AOAC (2000) guidelines. Nitrogen content was determined, and protein content calculated using the conversion factor 6.25 as in equation 3.

$$\%\ \text{N (wet)} = \frac{(\text{A} - \text{B}) \times 14007\text{x}\ 100}{\text{Weigh (g) of sample}} \tag{3}$$

A= volume (ml) std HCl × normality of std HCl
B= volume (ml) std NaOH × normality of std NaOH

**Crude fat determination using** Equation 4 to determined gravimetrically, using Soxhlet extraction. Approximately 5.0 g of sample was weighed into a pre-weighed extraction thimble ($W_1$). The thimble was placed in the Soxhlet apparatus, and extraction was performed using petroleum ether (60-80°C) for 3 hours. After extraction, the solvent was distilled off, and the flask containing extracted fat was dried in an oven at 100°C for 30 minutes. The cooled flask was reweighed ($W_2$).

$$\% \text{ Fat} = \frac{\text{Weight of lipid} \times 100}{\text{Weigh (g) of sample}} \quad (4)$$

**Crude fiber determination:** Fat-free residue from crude fat determination was subjected to sequential digestion with dilute sulfuric acid (1.25%) and dilute sodium hydroxide solution (1.25%) under reflux conditions. The residue was filtered, washed, dried, weighed, ashed, and reweighed. The difference represents crude fiber content.

**Total carbohydrate determination:** Total carbohydrate content was calculated by difference:
Total carbohydrate = 100 − (% lipid + % ash + % moist + % protein) (5)

### 2.3.2 BET (*Brunauer-Emmet-Teller*) surface area analysis

Surface area, pore volume, and pore size distribution were determined using a Micromeritics TriStar 3000 V4.02 analyzer employing nitrogen adsorption-desorption isotherms at 77 K. Approximately 0.3 g of each catalyst sample was accurately weighed and loaded into BET glass sample tubes. Samples were degassed at 473 K (200°C) for 3 hours using a Micromeritics FlowPrep 067 unit connected to high-purity nitrogen gas to remove physiosorbed water and other contaminants. Degassed samples were reweighed, and the actual sample mass used for analysis was recorded. BET surface area was calculated from the nitrogen adsorption isotherm in the relative pressure range of 0.05 to 0.30. Pore volume and pore size distribution were determined using the Barrett-Joyner-Halenda (BJH) method from the desorption branch of the isotherm.

### 2.3.3 Scanning electron microscopy and energy dispersive X-ray spectroscopy (SEM and EDX)

Morphological characterization and elemental analysis were performed using a Hitachi SU 3500 scanning electron microscope equipped with an EDX detector. Sample preparation involved mounting small amounts of catalyst powder onto aluminum stubs using double-sided carbon tape. Samples were sputter-coated with a thin gold layer (~20 nm thickness) under vacuum using a gold sputter coater to enhance conductivity and prevent charge accumulation during electron bombardment. SEM imaging was conducted at various magnifications (500× to 50,000×) using an accelerating voltage of 15-20 kV and working distance of 10-15 mm. Secondary electron (SE) imaging mode was employed to obtain surface topography information. EDX analysis was performed on multiple regions of each sample to determine elemental composition, with particular focus on carbon, oxygen, iron, sulfur, silicon, and calcium content. Quantitative elemental analysis was conducted using standardless ZAF correction methods.

### 2.3.4 Fourier transform infrared spectroscopy (FTIR)

FTIR analysis was performed to identify functional groups present in the catalysts. Spectra were recorded in the range of 4000-400 $\text{cm}^{-1}$ using the KBr pellet method.

## 3. RESULTS AND DISCUSSION

### 3.1 Proximate Analysis

Table 1 presents the proximate composition of all catalyst samples. Moisture content ranged from 8.70% (Sample O) to 10.95% (Sample P), indicating successful drying and minimal hygroscopic water retention. Crude protein content varied significantly, with Sample P exhibiting the highest value (19.72%) and Sample Q the lowest (5.47%). This variation reflects differences in the original starch sources and the extent of protein denaturation during calcination.

**Table 1:** Proximate Analysis of Biopolymer-Derived Catalysts

| Sample | Description | Moisture (%) | Crude Protein (%) | Crude Fat (%) | Crude Fiber (%) | Ash (%) | Carbohydrate (%) |
|---|---|---|---|---|---|---|---|
| M | Mixed (calcined) | 10.21 | 15.06 | 1.83 | 0.62 | 1.42 | 71.48 |
| N | Mixed (Fe-impregnated) | 9.35 | 17.20 | 2.50 | 2.01 | 2.11 | 68.84 |
| O | Corn (Fe-impregnated) | 8.70 | 7.15 | 3.21 | 1.48 | 1.20 | 79.74 |
| P | Corn (Fe-impregnated) | 10.95 | 19.72 | 4.18 | 2.75 | 3.27 | 61.88 |
| Q | Rice (Fe-impregnated) | 10.26 | 5.47 | 0.75 | 3.10 | 1.01 | 82.51 |
| R | Rice (Fe-impregnated) | 10.09 | 6.13 | 0.68 | 2.51 | 0.74 | 82.36 |

Crude fat content was generally low across all samples (0.68-4.18%), with corn-based catalysts showing slightly higher lipid retention. Crude fiber content ranged from 0.62% to 3.10%, with rice starch samples displaying higher fiber content due to the presence of cellulosic material from rice husks. Ash content remained relatively low (0.74-3.27%), suggesting efficient organic matter combustion during calcination. Total carbohydrate content was highest in rice starch catalysts (>82%), indicating substantial retention of polysaccharide-derived carbon framework after thermal treatment.

### 3.2 BET Surface Area Analysis

BET analysis revealed significant variations in surface area, pore volume, and pore diameter among the catalyst samples shown in Table 2. All samples were degassed at 473 K for 4 hours prior to analysis, ensuring removal of physiosorbed species.

Sample R (rice starch, Fe-impregnated) exhibited the highest surface area (270.326 $m^2/g$), followed by Sample M (208.528 $m^2/g$). The high surface area of Sample R can be attributed to the extensive pore structure development during calcination of rice starch matrix. Interestingly, Samples Q and R showed contrasting pore characteristics: while Sample R possessed higher surface area with smaller pore volume, Sample Q displayed larger pore diameter (11.614 nm) with significantly lower pore volume (0.0531 $cm^3/g$). This suggests different pore network architectures arising from variations in the calcination behavior of rice starch.

Mixed starch samples (M and N) demonstrated intermediate surface areas (175-208 $m^2/g$) with consistent pore volumes (0.826-0.863 $cm^3/g$) and small pore diameters (<2 nm), characteristic of microporous materials. Corn starch samples (O and P) showed moderate surface areas (137-

162 m²/g) with the highest pore volumes among all samples (0.892-0.926 cm³/g), indicating well-developed mesoporous structures.

The predominantly mesoporous nature of most catalysts (pore diameters 1.2-1.9 nm) is advantageous for biodiesel production, as it facilitates efficient mass transfer of triglyceride molecules during transesterification reactions.

**Table 2:** BET Surface Area Parameters

| Sample | Surface Area (m²/g) | Pore Volume (cm³/g) | Pore Diameter (nm) |
|---|---|---|---|
| M | 208.528 | 0.826 | 1.872 |
| N | 175.063 | 0.863 | 1.205 |
| O | 137.410 | 0.892 | 1.943 |
| P | 162.391 | 0.926 | 1.248 |
| Q | 70.895 | 0.0531 | 11.614 |
| R | 270.326 | 0.0190 | 6.508 |

### 3.3 SEM-EDX Analysis

### 3.3.1 Morphological characterization

SEM analysis revealed diverse morphological features across the catalyst samples, reflecting the influence of starch source and processing conditions on final particle architecture.

**Sample M (Mixed starch, calcined):** Exhibited a granular and angular appearance with clustered morphology. Particles displayed irregular shapes with sizes ranging from 5-20 µm. The clustering suggests partial agglomeration during calcination. Height: 21 mm, Bulk density: 0.68 g/cm³.

**Sample N (Mixed starch, Fe-impregnated):** Demonstrated polyhedral forms with spherical granules featuring interconnected macropores. The presence of macropores indicates gas evolution during organic matter decomposition. Particle size distribution was relatively uniform (10-25 µm). Height: 22 mm, Bulk density: 0.74 g/cm³.

**Sample O (Corn starch, Fe-impregnated):** Characterized by irregular particle shapes with agglomerated micro-spheres possessing smooth surfaces. The smooth surface texture suggests complete thermal decomposition of organic components. Average particle size: 8-18 µm. Height: 22 mm, Bulk density: 0.54 g/cm³.

**Sample P (Corn starch, Fe-impregnated):** Featured polygonal granules with smooth surfaces and high regularity. This regular morphology indicates uniform calcination conditions and consistent particle formation. Particle size: 12-30 µm. Height: 23 mm, Bulk density: 0.58 g/cm³.

**Sample Q (Rice starch, Fe-impregnated):** Displayed irregular shapes with interconnected dendritic structures. The dendritic morphology suggests complex pore network formation during thermal treatment. Particle size varied widely (5-35 µm). Height: 20 mm, Bulk density: 0.70 g/cm³.

**Sample R (Rice starch, Fe-impregnated):** Exhibited predominantly polyhedral shapes with irregular, angular, and smooth surfaces. This sample showed the most developed structure with well-defined particle boundaries. Particle size: 15-40 µm. Height: 24 mm (highest), Bulk density: 0.74 g/cm³ (highest).

The SEM observations indicate that calcination at 800°C effectively transforms the starch biopolymer matrix into diverse porous structures. The morphological differences between rice and corn-based catalysts suggest that the amylose/amylopectin ratio and residual protein/fiber content influence final particle architecture.

### 3.3.2 Elemental composition (EDX Analysis)

EDX spectroscopy confirmed successful iron incorporation across all catalyst samples and provided quantitative elemental analysis in Table 3.

**Table 3:** Elemental Composition from EDX Analysis

| Element | M (%) | N (%) | O (%) | P (%) | Q (%) | R (%) |
|---|---|---|---|---|---|---|
| Carbon (C) | 69.15 | 66.31 | 75.00 | 74.10 | 65.08 | 63.27 |
| Oxygen (O) | 16.70 | 14.18 | 10.24 | 12.06 | 8.75 | 12.34 |
| Iron (Fe) | 12.45 | 15.08 | 14.00 | 13.15 | 20.84 | 18.45 |
| Sulfur (S) | 0.52 | 2.01 | 0.13 | 0.14 | 1.36 | 1.08 |
| Silicon (Si) | 0.63 | 0.47 | 0.36 | 0.41 | 2.15 | 2.80 |
| Calcium (Ca) | 0.38 | 1.91 | 0.20 | 0.12 | 1.77 | 1.96 |

**Key Observations:**

1. **Iron Content:** Rice starch-based catalysts (Q and R) demonstrated the highest iron content (20.84% and 18.45%, respectively), indicating superior metal uptake capacity. Mixed and corn starch catalysts showed iron content ranging from 12.45% to 15.08%. The successful iron incorporation across all samples confirms the effectiveness of the wet impregnation method.
2. **Carbon Content:** Corn starch catalysts (O and P) exhibited the highest carbon content (~75%), suggesting greater retention of carbonaceous framework after calcination. This carbon-rich matrix can enhance catalyst stability and provide additional surface sites for catalytic activity.
3. **Oxygen Content:** Varied from 8.75% (Sample Q) to 16.70% (Sample M). Higher oxygen content in mixed starch samples may indicate the presence of metal oxide species and residual hydroxyl groups.
4. **Sulfur Content:** Ranged from 0.13% to 2.01%, with Sample N showing the highest sulfur retention. Sulfur originates from the Iron(II) sulphate impregnating agent and may contribute to acid site formation.
5. **Minor Elements:** Silicon (0.36-2.80%) and calcium (0.12-1.96%) were detected, likely originating from the agricultural waste biomass. Rice starch catalysts showed higher silicon content, possibly from silica present in rice husks.

The elemental composition data confirm that the biopolymer-derived catalysts possess a carbon-supported iron oxide structure, which is favorable for heterogeneous catalysis applications. The presence of multiple elements (C, O, Fe, S) suggests potential for multifunctional catalytic activity. Present your findings clearly and objectively using figures, tables, and equations to support your results. All figures should be numbered serially and placed at the referenced point with captions at the bottom (centralized). Further, interpret and discuss the significance of your results. Compare findings with previous research and explain any discrepancies. The results obtained are consistent with the theoretical predictions (Wilson et al., 2023; Ahmed, 2024a, 2024b).

The experimental results are demonstrated here (Author, 2024). As shown in Figure 1, the tensile strength increased substantially with quenching treatment, reaching a maximum of 680 ± 25 MPa, while elongation decreased correspondingly. Table 1 presents the measured parameters and their statistical variations.

## 4. CONCLUSION

This study successfully demonstrates the feasibility of utilizing biopolymer substrates, specifically rice and corn starch from agricultural waste, as precursors for developing heterogeneous catalysts for biodiesel production. The following key conclusions can be drawn:

1. **Successful Catalyst Synthesis:** The wet impregnation method followed by calcination at 800°C effectively transforms starch biopolymers into porous, iron-containing heterogeneous catalysts with diverse morphological and chemical properties.
2. **Superior Rice Starch Performance:** Rice starch-based catalysts, particularly Sample R, exhibited the highest surface area (270.326 $m^2/g$), bulk density (0.74 $g/cm^3$), and iron content (18.45%), indicating superior structural development and metal incorporation capacity.
3. **Favorable Structural Properties:** All catalysts demonstrated mesoporous characteristics with pore diameters ranging from 1.2 to 11.6 nm, suitable for accommodating triglyceride molecules during transesterification reactions. Surface areas (70-270 $m^2/g$) are comparable to effective biodiesel production catalysts reported in literature.
4. **Successful Iron Incorporation:** EDX analysis confirmed iron content ranging from 12.45% to 20.84%, demonstrating effective metal impregnation across all starch types. The presence of carbon (63-75%), oxygen (8-17%), and iron provides multifunctional active sites.
5. **Morphological Diversity:** SEM imaging revealed diverse particle morphologies including granular, polyhedral, spherical, and dendritic structures, reflecting the influence of starch source and composition on thermal decomposition behavior.
6. **Environmental and Economic Viability:** The utilization of agricultural waste as catalyst precursors addresses multiple sustainability objectives: waste valorization,

resource efficiency, reduced environmental impact, and cost-effectiveness through simple synthesis procedures.

7. **Green Chemistry Approach:** The catalyst preparation process aligns with green chemistry principles by minimizing hazardous chemicals, utilizing renewable resources, and producing minimal waste.

The present research establishes biopolymer substrates as promising, sustainable alternatives to conventional catalyst precursors for biodiesel production. The characterized catalysts possess appropriate surface properties, chemical composition, and morphological features conducive to heterogeneous catalysis applications. Rice starch, in particular, demonstrates exceptional potential due to its superior surface area development and metal incorporation capacity.

Future work should focus on validating catalytic activity through actual transesterification experiments, optimizing reaction conditions, assessing catalyst stability and reusability, and conducting comprehensive techno-economic analyses to facilitate industrial implementation. The findings contribute to the growing body of knowledge on sustainable catalyst development and support the transition toward circular economy principles in renewable energy production.

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