Utilization of polystyrene waste with biodiesel from cooking oil waste as feedstock in catalytic cracking using Al-MCM-41/Ceramic and Pd/Al-MCM-41/Ceramic catalysts

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Abstract. Liquid fuel from polystyrene plastic waste with co-reactant of cooking oil waste biodiesel has been obtained. The catalysts used in catalytic cracking are Al-MCM-41/ceramic and Pd/Al-MCM-41/ceramic catalysts. Morphology and elemental composition were observed with SEM EDX, the crystal structure observed with XRD, surface area and pore volume were analyzed by Nitrogen adsorption-desorption each calculated by BET and BJH method, acidity determined by FTIR-Pyridine, and temperature resistance analyzed by DTA. The results of catalytic cracking were investigated using Gas Chromatography-Mass Spectroscopy (GC-MS). Characterization of the catalyst showed that acidity was reduced because the Si/Al ratio was reduced after the impregnation and the surface area of Al-MCM-41/ceramics also decreased. SEM EDX shows that the composition of the carbon element on the surface of the Pd/Al-MCM-41/ceramic catalyst is smaller compared to the Al-MCM-41/ceramic catalyst. The results of the GC-MS characterization of liquid fuels from catalytic cracking using Pd/Al-MCM-41/ceramics catalyst, have a percentage of gasoline fraction (C7-C12) of 74.9% at 120 minutes cracking. The mixture of fuel from the addition of 150 mL liquid fuel resulting from catalytic cracking with Pd/Al-MCM-41/ceramic catalyst has the highest calorific value of 19160.61 (kcal/kg) and the performance of the gasoline Genset engine has the highest thermal efficiency of 28.27%.

1. Introduction

Polystyrene (PS) is a thermoplastic polymer compound with a linear hydrocarbon chain. Problems found in polystyrene waste are additives such as stabilizers and dyes. Additives can contain various poisons, especially cadmium and lead. Plastics can reach about 28% of total cadmium in municipal solid waste in studies at landfills [1]. Polystyrene recycling is needed and the road is cost-effective and environmentally friendly in processing plastics. Recycling PS plastic will help maintain the source of oil/petroleum because PS is a product based on petroleum. The thermal treatment of polystyrene plastic waste shows a useful option for reducing the amount of waste in Final Disposal Sites [1]. The thermal treatment can produce liquid fuel which is used as an alternative fuel source.

The use of biodiesel co-reactant into liquid fuels from polystyrene waste can increase the long chain hydrocarbon fraction, so that it can increase fuel resistance in the vehicle. Biomass catalytic cracking or co-catalytic cracking with plastic has been recommended to improve the quality of
biofuels [2]. Biodiesel is generally derived from plant oils or vegetable oils and also from animal fats obtained through the esterification and transesterification process. This oil can also be produced by utilizing household waste, which is cooking oil waste. Cooking oil waste has a large enough potential to be developed into biodiesel fuel because it has high fatty acids [3]. The catalytic cracking process from the co-reactant mixture of cooking oil waste and polystyrene biodiesel was carried out with MCM-41 catalyst. Catalytic cracking is more efficient than thermal cracking because the process only requires a fairly low energy due to the presence of a catalyst. One type of cracking process that converts FAME compounds into hydrocarbon fractions is by the active site of acid in the catalyst structure [4].

One of the catalysts that can be used for catalytic cracking in mixed polystyrene waste with co-reactant of cooking oil waste biodiesel, is Al-MCM-41. Al-MCM-41 is a mesoporous catalyst that has high acidity and is effective in catalytic cracking due to the presence of aluminum metal which can increase acidity. Acidity can affect the hydrocarbon fraction of the results of catalytic cracking. The acidity value can also be increased by increasing the composition of the metal. Ceramics are a source of Al metal that can increase the acidity of Al-MCM-41. In addition, ceramics can also increase the pore volume of the Al-MCM-41 catalyst because there is a solid-solid attraction between the surface of the catalyst and ceramics [5]. Adding transition metals can also increase acidity. One of the metals used in this study is Palladium metal (Pd). The catalytic ability of Pd metals is influenced by electrons in d orbitals that blend with the electrons of the s orbitals and the closest p degenerates. Blank orbitals in Pd metal can be used as metal catalytic sites. Metals tend to play a role in the dehydrogenation process and can help increase the yield of cracking based on the mechanism of hydrocarbon cracking reactions because the dehydrogenation mechanism makes it possible to break C-C bonds [6]. Cracking biodiesel with Pd/Al-MCM-41 catalyst can produce more hydrocarbons than alcohol [7].

This study conducted the performance of the products of polystyrene waste cracking with co-reactant of waste cooking oil biodiesel using Al-MCM-41/ceramic catalyst and Pd/Al-MCM-41/ceramic catalyst. The influence was obtained from the evaluation results through characterization on both catalysts using XRD, SEM-EDX, DTA, FTIR-Piridin and N₂ Adsorption-Desorption. GC-MS and the performance test on the products of cracking using the Generator Set engine.

2. Materials and methods

2.1. Material
The materials used in this study are household cooking oil waste, polystyrene plastic waste (PS), Al-MCM-41 catalyst obtained from previous studies [8], solids PdCl₂ (59% anhydrous Pd) from Sigma, HCl solution (38%) from Merck, Nitrogen UHP, Hydrogen UHP and Methanol p.a. from Merck

2.2. Production of liquid fuel from polystyrene plastic waste
Liquid fuel production is carried out through a thermal cracking process at 300 °C for 2 hours with continuous reactor shown in Figure 1. Liquid fuel from PS plastic is stored in vial bottles

2.3. Production of biodiesel from cooking oil waste
The biodiesel synthesis was carried out in a reflux reactor at 50°C and a speed of 700 rpm for 2 hours where cooking oil waste was heated at 50°C then added a mixture of 0.5% (w/w) sodium hydroxide and methanol.

2.4. Synthesis of Pd/Al-MCM-41/Ceramic catalyst
The Pd/Al-MCM-41/ceramic catalyst was made by hydrothermal technique using the wet impregnation method [8]. In this study, the impregnation method was carried out by hydrothermal technique using HCl solvents. Al-MCM-41/ceramic catalyst is included in 1% (w/w) of PdCl₂ solution. Mixing is done at room temperature and stirred evenly and slowly for 6 hours to evaporate the solvent so that solid uniformity is obtained. Calcination was carried out using a furnace at 500 °C for 1 hour.
The catalyst is reduced by flowed hydrogen gas. Then heated at a temperature of 150ºC for 1 hour. Pd/Al-MCM-41/ceramic catalyst is stored in the desiccator.

2.5. Characterization of catalyst
Pd metal impregnated in Al-MCM-41/ceramic catalyst was analyzed using XRD. The acidity of the sample was determined by the absorption of pyridine and analyzed using FTIR. SEM EDX is used to identify the morphology and elemental composition. Measurement of N₂ adsorption-desorption from calcined samples was carried out using Tristar II 3020. Calculation of surface area was carried out using the Brunauer-Emmet-Teller (BET) method and the pore volume was measured through the Barrett-Joyner-Halenda (BJH) method. The temperature resistance of the Pd/Al-MCM-41/ceramic catalyst was analyzed by DTA.

2.6. Preparation of catalyst
Al-MCM-41/ceramic catalyst was activated at 100 ºC. Pellet shaped of Al-MCM-41/ceramic is inserted into a catalyst tube filled with glass wool [9]. Pd/Al-MCM-41/ceramic was also activated at a temperature of 100 ºC and the pellet of catalyst was inserted into a catalyst tube filled with glass wool.

2.7. Catalyst activity
Liquid fuel from mixture of PS oil and co-reactant of cooking oil waste biodiesel is produced through catalytic cracking with Al-MCM-41/ceramic and Pd/Al-MCM-41/ceramic catalysts. The cracking is carried out in the continuous reactor shown in Figure 1 at 300 ºC for 2 hours. The results of catalytic cracking for 60 minutes and 120 minutes were analyzed by Gas Chromatography-Mass Spectrometer (GC-MS). Liquid fuel is mixed with Premium RON 88 and MTBE to obtain optimum performance on gasoline engines with the composition shown in Table 1.

![Figure 1. Continuous reactor scheme, 1. N₂ gas flow; 2. Feedstock flow; 3. Chamber Reactor; 4. Connector; 5. Condenser; 6. Oil tank; 7. GC-MS.](image)

| Catalyst         | Liquid Fuel Volume (mL) | Premium (88) (mL) | MTBE (mL) | Mixed Liquid Fuel |
|------------------|-------------------------|-------------------|-----------|-------------------|
| Al-MCM-41/Ceramic| 75                      | 750               | 18.4      | PMA1              |
|                  | 150                     | 750               | 18.4      | PMA2              |
| Pd/Al-MCM-41/Ceramic| 75                      | 750               | 18.4      | PMP1              |
|                  | 150                     | 750               | 18.4      | PMP2              |

2.8. Characterization of hydrocarbon from liquid fuel and performance test on gasoline engine
Hydrocarbon from liquid fuel are characterized by Gas Chromatography-Mass Spectrometer (GC-MS) to determine the composition of the hydrocarbon fraction from the results of the catalytic cracking of polystyrene oil with co-reactant of cooking oil waste biodiesel and heating value tests to be applied in the performance test. The performance test of the mixed liquid fuel was carried out using the
Generator Set (Gen Set) engine to determine the efficiency value of the mixture of liquid fuels against the load generated during the combustion process in the engine.

3. Results and discussion

3.1. Characterization of catalysts

The diffractogram of both catalysts was shown in Figure 2. The typical peak of Al-MCM-41 is still visible at 2θ between 20° and 30° even though Pd has been impregnated. The XRD pattern of the Pd/Al-MCM-41/ceramic catalyst shows that the impregnation of Pd does not damage the hexagonal structure of Al-MCM-41. However, the intensity of Al-MCM-41 after impregnation Pd falls. At 2θ: 25° shown in Figure 2(b) number 1 is a typical peak of crystals from Al. At 2θ: 33° and 41° shown in Figure 2(b) number 2 is the typical peak of the Pd crystal. The typical peak Pd is at 2θ: 34°; 42°; 55° and 62° [8]. At 2θ: 35°; 36°; 38° and 43° shown in Figure 2(b) number 3 is the top of the ceramic. Typical peak data of Pd showed that Pd metal has been impregnated on the Al-MCM-41/ceramic catalyst, but there is a peak shift due to the presence of ceramics.

![Figure 2](image2.png)

**Figure 2.** XRD Catalysts of (a) Al-MCM-41/ceramic (b) Pd/Al-MCM-41/ceramic.

![Figure 3](image3.png)

**Figure 3.** Micrograph SEM (a) Al-MCM-41/ceramic catalyst and (b) Pd/Al-MCM-41/ceramic catalyst.
Figure 4. SEM-EDX (a) Al metal on Al-MCM-41/ceramic catalyst (b) Al metal on Pd/Al-MCM-41/ceramic and (c) Pd metal on Pd/Al-MCM-41/ceramic catalyst.

The SEM microgram shown in Figure 3(a), shows the ceramic structure at point A forming a surface that looks flat on Al-MCM-41 and looks bright on the surface. The SEM microgram shown in Figure 3(b) which is a Pd/Al-MCM-41/ceramic catalyst, shows clumps at point A with a smooth surface such as clouds and flat and point B shows ceramics with a flat surface.

Impregnation of Pd metal does not affect the morphology of MCM-41, so there is no damage to the pattern. EDX analysis was carried out to determine the distribution of Pd metal in the Al-MCM-41/ceramic catalyst. Figure 4 is an EDX image of Al-MCM-41/ceramic and Pd/Al-MCM-41/ceramic catalysts. Figure 4(c) showed that the presence of Pd metal has been identified in the Al-MCM-41/ceramic catalyst evenly as can be seen in the results of EDX analysis in Table 2.

| Catalyst                  | Composition of Each Element (%) |
|---------------------------|---------------------------------|
|                           | O   | Si  | C   | Al  | Pd  | N   |
| Al-MCM-41/Ceramic        | 68.9| 10.16 | 20.21 | 0.73 | 0   | 0   |
| Pd/Al-MCM-41/Ceramic    | 9461| 3.4 | 1.42 | 0.43 | 0.14 | 0   |

The composition of Si and Al is reduced by 6.76% and 0.3% after Pd impregnation shown in Table 2. Aluminum metal as the active metal in the catalyst can be reduced due to leaching. The catalyst can be deactivated by the loss of the active component due to leaching [10]. Al metal clumping occurs in the Pd/Al-MCM-41/ceramic catalyst shown in figure 4(b) while the Al metal on the Al-MCM-41/ceramic catalyst spreads evenly on the surface of the catalyst shown in Figure 4(a). In addition, the composition of carbon in the Al-MCM-41/ceramic catalyst impregnated with Pd becomes smaller than Al-MCM-41/ceramics which is 1.42%. The results of N2 adsorption-desorption are shown in Figure 5. The isotherm graph of both catalysts is an isotherm graph of type IV where the pore structure in both catalysts is mesoporous. This form is a typical form of Al-MCM-41. Previous research [8] showed that the surface area and pore volume decreased in Al-MCM-41 catalyst when Pd metal was impregnated on the surface of the catalyst because the Pd metal was more adsorbed to the small pore mouth. Metal Pd can also increase the average finger pore because Pd is more absorbed in small pores and occupies other pore mouth regions [8]. Therefore, the role of Pd metal in the Al-MCM-41 catalyst is good for catalytic cracking.

The surface area of the Pd/Al-MCM-41/ceramic catalyst is smaller than that of the Al-MCM-41/ceramic catalyst because Al metal clumping occurs on the surface of the Pd/Al-MCM-41/ceramic catalyst shown in Table 3. Clumping can reduce the contact surface area between the active side of the catalyst and reactants [11]. Changes in pore texture can affect the performance of a catalyst by loss of specific surface area through sintering of the carrier [10]. Pore volume in Pd/Al-MCM-41/ceramic catalyst is higher than Al-MCM-41/ceramic. The process of catalytic cracking occurs in the pore of the catalyst and not on the surface [8]. Therefore, Pd/Al-MCM-41/ceramic catalysts can select oil gas well when cracking because there are many active sides of the pore catalyst.
Figure 5. Isotherm graph of (a) Al-MCM-41/ceramic catalyst and (b) Pd/Al-MCM-41/ceramic catalyst.

Table 3. Data on N2 adsorption-desorption analysis.

| Catalyst                  | Surface Area (m²/g) | Pore Volume (cc/g) | Pore Radii Average (Å) |
|---------------------------|---------------------|--------------------|------------------------|
| Al-MCM-41/Ceramic         | 490.665             | 0.546              | 22.22                  |
| Pd/Al-MCM-41/Ceramic      | 358.220             | 3.046              | 170.06                 |

The acidity of the catalyst was measured using pyridine adsorption analyzed by FTIR shown in Figure 6. The acidity of catalyst can affect the product of the catalytic cracking process where the higher the acidity, the shorter the hydrocarbon fraction produced will be [5,8,12]. The acidity of the Al-MCM-41/ceramic catalyst and Pd/Al-MCM-41/ceramic catalyst can be known by FTIR analysis using pyridine compounds. From the FTIR spectrum shown in Figure 6, the Al-MCM-41/ceramic catalyst reacted with pyridine has interactions between catalysts and pyridine compounds at wavenumbers 1431-1460 cm⁻¹ and 1533-1558 cm⁻¹. The interaction between the pyridine molecule and the Bronsted acid side on the surface will give rise to an absorption band at the wave number area of 1540-1545 cm⁻¹, while the interaction between the molecular probe and the Lewis acid side surface appears on the wavenumber absorption around 1440-1452 cm⁻¹ [13]. Peak at wavenumber 1496 cm⁻¹ is the adsorption band combined Lewis acid side and Bronsted acid side [14].

Figure 6. FTIR-Pyridine spectrum on the catalyst.

At wavenumbers 1431-1460 cm⁻¹ shows the active side of Lewis acid with the amount of 0.0311 (mmol/g) and at wavenumber 1533-1558 cm⁻¹ shows the active side of Bronsted acid with the amount
of 0.0342 (mmol/g) like in Table 4. The Pd/Al-MCM-41/ceramics catalyst also interacts with pyridine compounds in the same wavenumber range as between Al-MCM-41/ceramic catalyst and pyridine compound at wavenumbers 1435-1458 cm⁻¹ and 1531-1556 cm⁻¹. At wavenumber 1435-1458 cm⁻¹ shows the active side of Lewis acid and at wave number 1531-1556 cm⁻¹ shows the active side of Bronsted acid. The total acid is obtained from the sum of the active side of Lewis acid and the active side of Bronsted acid.

### Table 4. Acidity of catalyst with FTIR-Piridin.

| Catalyst Type of Acid | Wavenumber (cm⁻¹) | Amount of Acid (mmol/g) | Total Acid (mmol/g) |
|-----------------------|-------------------|-------------------------|---------------------|
| Al-MCM-41/Keramik    | Lewis             | 1431-1460               | 0.0311              |
|                       | 0.0311            | 0.0653                  |
|                       | Bronsted          | 1533-1558               | 0.0342              |
| Pd/Al-MCM-41/Keramik | Lewis             | 1435-1458               | 0.0154              |
|                       | 0.0154            | 0.0172                  |
|                       | Bronsted          | 1531-1556               | 0.0018              |

The acidity of the Pd/Al-MCM-41/ceramic catalyst decreases because the Si and Al metal elements decrease in composition after Pd impregnation based on EDX data in Table 2. Si/Al ratio on Al-MCM-41/ceramics catalyst impregnated Pd is small which is 7.9% shown in Table 5. Previous research [15] regarding the acidity of zeolite was explained that an increase in Si/Al ratio would also increase the acidity of zeolites, because in general the acidity of zeolites was determined by the presence of hydroxide groups bound to Aluminum or on Silicon sites Aluminum. In addition, clumping can also reduce the acidity of the Pd/Al-MCM-41/ceramic catalyst. Clumping can also cause a decrease in the acidity of a catalyst because it causes a decrease in the dispersion of metals in the carrier [16]. Active metals such as Al in the Al-MCM-41/ceramic carrier can increase acidity when dispersed evenly on the surface of the catalyst.

### Table 5. The composition of Si and Al on the catalyst.

| Catalyst Type of Acid | Si (%) | Al (%) | Rasio Si/Al |
|-----------------------|--------|--------|-------------|
| Al-MCM-41/Ceramic     | 10.16  | 0.73   | 13.9        |
| Pd/Al-MCM-41/Ceramic  | 3.4    | 0.43   | 7.9         |

The resistance of the catalyst to the cracking temperature observed with the DTA analysis that shown in Figure 7. The endothermic reaction occurs between 35-270°C in the Al-MCM-41/ceramic and Pd/Al-MCM-41/ceramic catalyst which indicates that there was a dehydration reaction and changes in composition of the catalyst where the CO₂ gas formation reaction occurs from carbon oxidation. The endothermic reaction which was characterized by a downward curved graph was the process of decomposition or release of organic elements such as CO₂ and water vapor contained in the raw material [17].

Both catalysts experience the same reaction, namely the endotherm at this temperature. The exothermic reaction occurs at 270-750 °C which can be called High Temperature Oxidation (HTO). This reaction showed that the catalyst was metastable where the catalyst changes to a stable structure (no chemical reaction/mass change occurred) on the catalyst. The DTA exothermic process indicates a decrease in enthalpy of the sample which indicates a change to a more stable structure [18,19]. Therefore, cracking at a temperature of 300 °C is the right temperature where the catalyst leads to a more stable structure.

### 3.2. Results from the catalytic cracking process

Liquid fuel from catalytic cracking of PS plastic waste oil and cooking oil waste biodiesel co-reactant with Al-MCM-41/ceramic and Pd/Al-MCM-41/ceramic catalysts characterized by GC-MS and chromatogram are shown in figure 8. Some peaks appear with a variety of intensities and peaks that appear more and more when cracking is getting longer. From the interpretation of several hydrocarbon
on mass spectra, we can identify the percentage of hydrocarbon fractions in each liquid fuel as shown in Table 6.

![Figure 7. Thermal analysis of catalyst with DTA.](image-url)

**Table 6. Percentage of hydrocarbon fraction from cracking product.**

| Catalyst                   | Time (Minutes) | % Hydrocarbon Fraction |
|---------------------------|----------------|------------------------|
|                           |                | < C7 | C7-C12  | >C12 |
| Al-MCM-41/Ceramic         | 60             | 0    | 32.84   | 67.14 |
|                           | 120            | 2.32 | 59.1    | 38.58 |
| Pd/Al-MCM-41/Ceramic      | 60             | 0    | 37.33   | 62.67 |
|                           | 120            | 5.98 | 74.59   | 19.43 |

The percentage of gasoline fraction (C7-C12) increases with the length of time in catalytic cracking and is inversely proportional to the percentage of long chain hydrocarbon fractions (> C12). The process occurs because along the length of time, the gas phase of hydrocarbons in the mixture will continuously react to the active side of the catalyst so that cracking will continue to occur. The results of the short chain hydrocarbon fraction from catalytic cracking are also influenced by the active side of the Pd/Al-MCM-41/ceramic catalyst more than the Al-MCM-41/ceramic catalyst shown in the EDX data in Table 2. The presence of carbon in the Al-MCM-41/ceramics catalyst can cause crust in the form of coke during the cracking process so that it covers the active side of the catalyst. The closure of the active side of this catalyst causes deactivation of the catalyst and decreases the yield of the product [11].

The results of calorific value of the mixture of liquid fuels are shown in Figure 9. The results showed that mixed premium/MTBE with liquid from catalytic cracking using Pd/Al-MCM-41/ceramic catalysts significantly increased when the liquid fuel volume was increased compared to the liquid fuel's addition of catalytic cracking using Al-MCM-41/ceramic catalyst. The calorific values of MTBE and mixed premium/MTBE from catalytic cracking using Pd/Al-MCM-41/ceramic catalysts increased by 99.73%, while with the addition of liquid fuel from catalytic cracking using Al-MCM-41/ceramic catalysts only increased by 26%. Based on the calorific value data showed that addition of 150 mL mixed fuel from catalytic cracking with Pd/Al-MCM-41/ceramic catalyst has the highest heat value of 19160.61 kcal/kg.

The results of thermal efficiency of mixed fuel are shown in Figure 10. The thermal efficiency of mixed fuels with liquid fuel addition from catalytic cracking with Pd/Al-MCM-41/ceramic catalyst increased more than the liquid addition of catalytic cracking with Al-MCM-41/ceramic catalyst as much as 1.26%. Based on the thermal efficiency data of the two catalysts, the highest percentage of thermal efficiency was found in the mixture of fuel from the addition of liquid fuel about 150 mL as a result of catalytic cracking with Pd/Al-MCM-41/ceramic catalyst, is 28.27%. The gasoline fraction of
liquid fuel from catalytic cracking with Pd/Al-MCM-41/ceramic catalyst is greater than the Al-MCM-41/ceramic catalyst, so the calorific value is large and affects the increase in thermal efficiency. Calorific values and octane numbers can affect the percentage of thermal efficiency of liquid fuels [20].

**Figure 8.** Chromatogram of GC-MS from cracking product of PS oil and co-reactant of cooking oil waste biodiesel with (a) Al-MCM-41/ceramic catalyst (b) Pd/Al-MCM-41/ceramic catalyst.

**Figure 9.** Calorific value of mixed fuel.

**Figure 10.** Thermal efficiency of mixed fuel.
4. Conclusion

Conclusions can be drawn from catalytic cracking on polystyrene waste with co-reactant of cooking oil waste biodiesel using Al-MCM-41/ceramic and Pd/Al-MCM-41/ceramic catalysts. The Si/Al ratio of Al-MCM-41/ceramic catalyst is higher than the Al-MCM-41/ceramic catalyst at 13.9%. However, Pd/Al-MCM-41/ceramic have a composition of carbon (C) smaller than Al-MCM-41/ceramics which is equal to 1.42%. The pore volume of Pd/Al-MCM-41/ceramic catalyst is greater than Al-MCM-41/ceramic, which is 3.046 (cc/g). The results of the thermal analysis with DTA of the two catalysts show that at the cracking temperature (300 °C) an exothermic reaction occurs where the catalyst structure becomes stable, so that both catalysts are resistant to that temperature. Gasoline hydrocarbon fraction (C7-C12) was obtained more in Pd/Al-MCM-41/ceramics than Al-MCM-41/ceramic catalyst which was 74.9% at 120 minutes in cracking. The mixture of fuel from the addition of liquid fuel from catalytic cracking with Pd/Al-MCM-41/ceramics as much as 150 mL had the highest heating value of 19160.61 (kcal/kg) and the highest thermal efficiency of 28.27%.

Acknowledgements

The authors gratefully acknowledge the funding support from ITS Local Scheme of Grants as well as Higher Education Directorate of Republic Indonesia for Financial Grant in 2019.

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