Effect of zeolite catalyst concentration on bio-fuel characteristics of corncobs (Zea mays l.) produced by pyro-catalytic method

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Abstract: One alternative energy source that can be renewed is the conversion of biomass into bio-fuel. This study aims to produce bio-fuel from corn cobs and determine effect of catalyst concentration on physical and chemical properties. The pyro-catalytic process was carried out at 400°C with 5%, 6% and 7% catalyst variations. Bio-fuel physical characterization tests include density, viscosity, and calorific value, while the analysis of hydrocarbon compound content uses GC-MS instrumentation. The results showed optimum biofuel volume was obtained at 7% catalyst variation that is 232 ml, while for physical and chemical characteristics the best results were obtained at 6% catalyst percentage with a density of 0.9636 gr / ml, viscosity 28.59 cP, and the calorific value is 10,350 kcal / kg. GC-MS analysis showed the content of Phenol, Furfural, acetic acid, Benzene, and Toluene compounds with a fuel percentage of 56.13% on a 6% catalyst. Based on the overall analysis that has been done shows that bio-fuel can be used as a substitute material for diesel or alternative energy substitute for conventional fuel.

1. Introduction
Petroleum is a non-renewable natural resource commonly used as fuel. Almost all activities carried out require fuel, for example in the transportation, industrial and household sectors, so that petroleum supplies in the world are increasingly depleting and prices are increasing [1]. Therefore, efforts are needed to reduce human dependence on petroleum as a non-renewable energy source. One alternative energy source that can be renewed is the utilization of biomass into bio-fuel. Biomass can be used as a renewable energy source because it is environmentally friendly and has abundant reserves [2].

One of the potential biomass to be used as biofuel is corncobs (Zea mays L.). Corncob contain hemicellulose 36%, cellulose 41%, and lignin 16% [3][4]. The process used to produce biofuels is pyrolysis. From this process organic steam, gas and charcoal are obtained [5]. Pyrolysis is a thermochemical decomposition that occurs without oxygen. To accelerate the reaction in the pyrolysis process, it is necessary to have a catalyst [6]. One type of catalyst that can be used is zeolite, aluminum silicate crystal with a three-dimensional network structure. Zeolite-based catalysts are very effective in reducing oxygen levels in bio-oil and play a role in the formation of aromatic bonds [7].
addition, zeolite is also able to reduce water content in bio-oil. Natural zeolite was chosen because it has abundant availability at low prices.

The main technologies used in the conversion process of biomass to energy are thermochemistry and biochemistry. Pyrolysis is a decomposition process of organic or inorganic material which produces the main products in the form of solid char and liquid and gas as a by-product [8-10]. The liquid (bio-oil) of pyrolysis products usually contains acids, alcohols, aldehydes, ketones, esters, heterocyclic derivatives and phenolic compounds [11-14]. Bio-oil is a high density oxygenated liquid which can be used as a substitute for liquid fossil fuels in several applications [15]. Bio-oil can be used as a substitute for diesel engines, a feedstock in conventional petroleum refineries and enhanced to obtain more valuable light hydrocarbon fuels [16,17]. Direct use of bio-oil as conventional fuel is not recommended because of its high viscosity, corrosiveness, instability and poor heating value [18,19]. Therefore, further research was developed with catalytic substitution to produce quality derivative fuel products similar to conventional fuels [20].

In this research, bio-fuel production from corn cobs will be carried out through a pyrolysis process using a zeolite catalyst. Pyrolysis temperature used in this study was 400 ℃ and zeolite catalyst variation was 5, 6, 7% of the mass of biomass. As a comparison, the pyrolysis process without catalyst will be carried out. With this research, it is hoped that it can produce bio-oil from corn cobs waste as a renewable energy source that is used as a substitute for fuel from petroleum whose availability is decreasing and can help increase the country's economic value.

| Table 1. Proximate, ultimate and component analysis (wt%) of us s of corncob. |
|---|---|---|---|---|
| **Proximate**<sup>b</sup> | wt% | **Ultimate**<sup>b</sup> | wt% | **Component analysis**<sup>c</sup> | wt% |
| Volatiles | 76.7 | C | 52.7 | Extractives | 8.10 |
| Fixed Carbon | 15.7 | H | 6.4 | Hemicellulose | 32.1 |
| Ash | 1.2 | N | 0.3 | Lignin | 13.1 |
| Moisture | 6.4 | O<sup>a</sup> | 40.6 | Cellulose | 52.9 |

<sup>a</sup> The oxygen content was calculated by difference.

<sup>b</sup> Weight percentage on dry basis.

<sup>c</sup> Extractive free basis.

2. **Experimental Section**

2.1. **Biomass Sample and Catalyst**
The corncob sample investigated in this study has been taken from the city of Buton in Southeast Sulawesi region. Before the experiments, the samples were dried, cutter and then prepared to obtain uniform particle sizes. Particle size range was between 0.5 mm < d<sub>p</sub> < 1 mm. Table 1 shows the main characteristics of the corncob used.

Natural zeolit was used as a catalyst in the experiments. The variation of catalyst concentrations investigated in the research is 5, 6 and 7%.

2.2. **Experimental Pyrolysis Setup**
The pyrolysis experiments were carried out using a fixed-bed reactor. The effect of catalyst concentration was observed on the final product yield in each experiment. 500 g of sample is put into the pyrolysis reactor and temperature is increased at a flow rate of 15 ℃ / minute to the final pyrolysis temperature of 400 ℃.

The reactor was heated externally by an electric furnace, with the temperature being controlled by an powersuply outside the bed. condenser made of thick stainless pipe along 350 cm with an inner diameter of 18 cm, then connected to the pyrolysis product container. The pyrolysis furnace has a power of 1000 W with a large enough inner dimension so that it can accommodate all parts of the reactor..

2.3. **Bio-oil characterization**
2.3.1. **Analytical GC-MS (Gas Chromatography-Mass Spectrometry).** The chemical identification of bio-fuel hydrocarbons was carried out by the GC-MS instrument using a DB-5 column (60 m x 0.25 mm x 0.25 μm) agilent, injector temperature was 280°C, and the carrier gas separation ratio was 69.4 the carrier gas was helium in the flow control of 1 mL/min. Initially, the oven temperature is kept at 45°C for 2 minutes, then the temperature rises to 280°C at 5°C / minute and held for 2 minutes.

2.3.2. **Analytical Physics of Bio-Fuel.** The fuel oil density was analyzed using a 25 ml picometer, then calculated by Eq. (1) and measurements are in accordance with ISO 4787 standards [21][22]. To minimize measurement errors, all measurements were carried out three times for each sample and the results were averaged.

\[ \rho = \frac{m}{V_p} \text{(g/mL)} \]  

Where:
\( m \): mass (pycnometer + sample) - empty pycnometer mass.
\( V_p \): Pycnometer volume (25 ml).

The fuel-oil viscosity is determined by comparing the viscosity of the fuel-oil and the viscosity of the comparative liquid in the form of water using an ostwald viscometer involving each of the fuel-oil density and water density variables, then calculated by equation (2) [21]:

\[ \frac{\eta_1}{\eta_2} = \frac{\rho_1 t_1}{\rho_2 t_2} \]  

Where:
\( \eta_1 \): Fuel-oil (cP) viscosity; \( \eta_2 \): Water viscosity (cP)
\( \rho_1 \): Fuel-oil density (g / ml); \( \rho_2 \): Water density (g / ml)
\( t_1 \): Fuel-oil (s) flow time; \( t_2 \): Water flow time (s)

Calorific value (C) is analyzed by involving the variable density, specific gravity and API (American Petroleum Institute) gravity. Specific gravity (SG) is the ratio between fuel-oil density and water density, while the API gravity value is calculated using the following equation[21][23]:

\[ ^oAPI = 141.5 \div SG - 131.5 \]  

\[ C = \frac{2.2046226}{3.9673727} \times (18.650 + 40 \times (SG - 10)) \text{ kcal/kg} \]  

3. **Results and Discussion**

3.1. **Influence of Pyrolysis Parameters on The Product Yields**

Bio-fuel is a black-colored liquid fuel derived from biomass [24]. Bio-fuel can be obtained through the pyrolysis process. Bio-fuel has a standard color from dark green to dark red to black depending on the materials and processes used [25].

Based on observations that have been made show that the variation of the percentage of zeolite catalysts affects the temperature of the formation of pyrolysis liquid. Before the temperature of 400°C the pyrolysis liquid was formed, namely in the 5% zeolite catalyst the pyrolysis liquid was formed at 378°C, then the 6% zeolite catalyst was formed at 360 °C and the 7% catalyst was formed at 353°C. The more catalysts used, the temperature of the formation of pyrolysis liquids will be lower. This happens because the catalyst will increase the reaction rate, which causes the activation energy used in the reaction process to be lower, so the reaction or cracking of hydrocarbons will take place quickly [26].

Increasing the catalyst percentage also affects the pyrolysis time. According to Fatimah [13][27] zeolite in the pyrolysis process will provide a fast and stable temperature propagation in the biomass due to the presence of alumina in the zeolite, so that with the process the temperature will break the
chemical structure of the biomass, besides that the process of breaking up hydrocarbons occurs faster due to the presence of the role of pore structure in zeolites. At the same time zeolite also acts as a cation exchange where this process will occur on the surface and inside the zeolite pores. Zeolite will stimulate molecules charged in pyrolysis product gas, first zeolite will attract protons from pyrolysis product gas due to potential differences. Pyrolysis product gas has a more positive potential while zeolite is more negative, where the negative zeolite is caused by aluminum content. With this there is a potential difference that protons move from the pyrolysis product gas to the zeolite so that the pyrolysis product gas lacks protons or excess electrons. So that the molecules in the pyrolysis product gas will break easily and be more active. This causes the pyrolysis time on the zeolite catalyst to be 7% faster compared to the zeolite catalyst 6%, 5% and without the catalyst.

Figure 1. Correlation of zeolite catalyst variation with volume of bio-fuel from corncob

Figure 1. shows the relationship of zeolite catalyst variation with the volume of bio-fuel from corncobs. Based on the figure, it can be seen that the variation in the percentage of zeolite catalyst influences the volume of bio-fuel produced. The lowest bio-fuel volume was obtained in the absence of a catalyst (0%) while the highest volume was obtained under conditions using a 7% zeolite catalyst. In theory, the greater the amount of catalyst used, the greater the number of active surfaces, or where the pyrolysis reaction takes place, so that the resulting product is also increasingly large [26]. Zeolite has a very regular crystalline shape with interconnected cavities in all directions that cause a very large zeolite surface area, therefore the more amount of catalyst used will expand its active surface as a place for the reaction process or hydrocarbon cracking. The same thing was also said by Lestari [28], that by increasing the zeolite catalyst weight percentage to biomass with the same biomass size, the number of pores on the catalyst increased and the surface area also increased, so that the active sites on the catalyst that could be utilized during the cracking process more and more and can cause the volume of bio-fuel to increase. The amount of percentage of natural zeolite catalysts used, will increase possibility of reaction decomposition of cellulose, hemicellulose and lignin in the catalyst cavity causing the Bio-oil yield to be even greater [29].

3.2. Characterization of Chemical Properties of Bio-fuel

Characterization of the chemical properties of bio-fuel is carried out using Gas Chromatografy-Mass Spectrometry (GC-MS). GC-MS analysis is carried out to determine the types and composition of compounds contained in the products produced from the pyrolysis process. Each peak in the chromatogram is a signal that is produced when a compound is detected by a detector. GC-MS has become a quick and powerful tool for characterizing complex and heterogeneous bio-fuel samples [30]. There are various types of compounds identified in bio-fuel products which are classified as hydrocarbons, furfural, benzene, toluene and acetic acid. Table 2 shows a detailed component analysis of biofuels using GC-MS, including compound names, and chromatogram peak areas for all catalyst concentrations. In the above table, with respect to the aliphatic percentage, it was found to be higher with the addition of a catalyst. Because aliphatic is an important compound in terms of its similarity to fuel.
Table 2. The dominant compound from the GC-MS analysis of bio-oil from corn cobs with variations in the percentage of zeolite catalysts 0%, 5%, 6% and 7%.

| No | Compound Name                      | Formula      | Area (%)       |
|----|------------------------------------|--------------|----------------|
|    |                                     |              | 0%  | 5%  | 6%  | 7%  |
| 1  | Furfural                           | C₅H₆O₂      | 2.54 | 9.71| 6.58|     |
| 2  | Phenol, 2-methoxy-                 | C₇H₁₀O₂     | 9.52 | 7.58| 8.03|     |
| 3  | 1,6-Heptadien-4-ol                 | C₁₃H₁₆O₅    | 1.72 | 5.41| 0.59|     |
| 4  | Catechol                           | C₇H₆O₂      | 4.28 | 4.04| 5.19|     |
| 5  | 3-Methyleclopentane-1,2-dione      | C₁₀H₁₈O₃    | 2.6  | 3.96| 2.93|     |
| 6  | Phenol                             | C₆H₁₂O            | -    | 3.22| 2.85|     |
| 7  | D-Allose                           | C₈H₁₂O₈      | -    | 3.21| -   | -   |
| 8  | 2-Propanone, 1-(acetoxy)-          | C₆H₁₀O₃     | 1.11 | 1.15| 2.11|     |
| 9  | 1-Hydroxy-2-butane                 | C₅H₁₀O₂     | 1.95 | 3.13| 1.26|     |
| 10 | Phenol, 3-methyl-                  | C₆H₁₀O      | -    | -   | 1.16|     |
| 11 | Ethanone, 1-(2-furanyl)-           | C₅H₁₀O₂     | 1.31 | 1.65| 1.84|     |
| 12 | Creosol                            | C₁₇H₂₂O₂    | 0.51 | 1.20| 2.24|     |
| 13 | 1,3-Benzenediol, 2-methyl-         | C₇H₁₂O₂     | 1.51 | 1.57| 2.67|     |
| 14 | 1,3-Benzenediol, 4-ethyl-          | C₉H₁₆O₂     | 0.72 | 0.82| -   | -   |
| 15 | 1,2-Benzenediol, 3-methoxy-        | C₇H₁₀O₃     | -    | 0.36| -   | -   |
| 16 | 2,4-Dimethoxyphenol                | C₈H₁₀O₃     | -    | -   | 1.50|     |
| 17 | 2-Furancarboxaldehyde, 5-methyl-   | C₅H₁₀O₂     | 0.73 | -   | 1.66|     |
| 18 | 2-Furanol, tetrahydro-             | C₇H₁₀O₂     | -    | 0.30| 0.54|     |
| 19 | 3,5-Dimethoxy-4-hydroxytoluene     | C₉H₁₄O₃     | 0.76 | -   | 1.69|     |
| 20 | 2(5H)-Furanone                     | C₉H₁₂O₂     | 0.97 | 0.48| 1.57|     |
| 21 | 2-Furannethanol, tetrahydro-       | C₉H₁₂O₂     | -    | -   | 1.96|     |
| 22 | Phenol, 2,6-dimethoxy-             | C₉H₁₂O₃     | 3.29 | 2.74| 3.71|     |
| 23 | Phenol, 4-ethyl-2-methoxy-         | C₁₀H₁₄O₂    | 3.97 | 2.63| 2.47| 2.41|
| 24 | 2-Cyclopenten-1-one, 3-ethyl-2-hydroxy- | C₁₀H₁₄O₂ | 2.35 | 2.36| 2.80|     |
| 25 | Phenol, 4-ethyl-                   | C₈H₁₂O      | 3.51 | 2.25| 3.71| 2.19|
| 26 | 1,4,3,6-Dianhydro-a-d-glucopyranose| C₈H₁₄O₄     | 1.67 | 2.05| -   | -   |
| 27 | Ammonium acetate                   | C₅H₇NO₂     | -    | -   | 6.66|     |
| 28 | Acetic acid                        | CH₃COOH      | 5.29 | -   | 4.66|     |
| 29 | 3-Methyleclopentane-1,2-dione      | C₁₀H₁₄O₂    | 2.6  | -   | 2.93|     |
| 30 | Phosphonic acid, (p-hydroxyphenyl)-| C₈H₁₆O₅P    | 2.83 | -   | 2.16|     |
| 31 | 10-Octadecenoic acid, methyl ester | C₁₉H₃₈O₂    | -    | -   | -   | 34.46|

Table 3. Identification of the constituent compounds of pyrolysis bio-fuels

| No | Compound Name                      | Area (%)       |
|----|------------------------------------|----------------|
|    |                                     | 0(%) | 5(%) | 6(%) | 7(%) |
| 1  | Phenol                             | 23   | 26.58| 32.96| 4.6  |
| 2  | Furfural                           | 4.57 | 13.74| 14.15| -    |
| 3  | Benzene                            | 2.23 | 2.75 | 2.67 | 2.49 |
| 4  | Toluene                            | 0.76 | -    | 1.69 | -    |
| 5  | Acetic acid                        | 5.29 | -    | 4.66 | -    |
|    | Total (%)                          | 35.86| 43.07| 56.13| 7.09 |
Table 3 shows the identification of the constituent compounds of pyrolysis bio-fuel fuels consisting of 5 main compounds namely phenol, furfural, benzene, toluene, and acetic acid. Acetic acid or better known as vinegar acid (CH₃COOH) is a liquid, colorless, pungent compound, has a sharp acidic taste and dissolves in water, alcohol, glycerol, ether. At atmospheric pressure, the boiling point is 118.1 °C. Acetic acid has a very broad application in the industrial and food fields [31]. Furfural is a colorless liquid that is an organic compound from the furan group. Furfural in industries, among others, are used for oil processing, nylon making, resin making, pharmaceuticals, and others [32]. Bio-oil also contains up to thousands of different components, which are divided into five main categories, namely alcohol, phenols, ketones, aldehydes, and organic acids [33]. Acids, aldehydes and organic acids are products of cellulose and hemicellulose from biomass, while phenol is a product of lignin [34].

Benzene is an organic chemical compound in the form of a colorless and flammable liquid. Benzene is one of the components in petroleum, and is one of the most basic petrochemical ingredients and solvents that are important in the industrial world. Benzene has a high octane number, so benzene is also used as an important mixture in gasoline [35]. Also detected also toluene compounds which are benzene derivatives. Toluene also known as methylbenzene or phenylmethane, is a colorless, flammable liquid, insoluble in water solvents [36].

Based on the identification of the compounds making up the fuel, it was seen that there was a decrease in acid / acid group compounds and an increase in phenol group compounds as the percentage of zeolite catalyst increased. Decreased acid group/acid even disappear due to the decarbonylation reaction that breaks the oxygen bonds in the acid group so that it becomes an alkane and alkene chain. Meanwhile zeolite also acts as a catalyst for the formation of aromatic clusters by binding to gases such as ethylene, etane, propylene and propane through aromatization reactions to form aromatic clusters. Therefore, the phenol component is increased by pyrolysis involving zeolites as a catalyst. Phenol is an aromatic compound that has an OH chain [37].

![Figure 2. Percentage of fuel compounds](image)

Figure 2 shows the percentage of the optimum fuel constituent obtained in the 6% zeolite catalyst variation with a percentage of 56.13%. The addition of the amount of zeolite catalyst causes an increase in the composition of the fuel constituent compounds, but in the variation of the zeolite catalyst 7% the percentage of the fuel constituent compounds decreases, this is due to the use of too many catalysts causing the catalyst to be deactivated by reactants and abundant products, so that it can cover the cycle active on the catalyst. The use of too much catalyst can also cause molecules that have been formed to react again into heavier molecules [38].

3.3. Analytical Physics of Bio-Fuel

Characterization of physical properties performed includes determining density, viscosity, specific gravity, API gravity, and calorific value. Data from the analysis can be seen in Table 4.
Table 4. The results of the analysis of the physical properties of bio-fuel

| Zeolite catalyst (wt%) | Density (g/ml) | Viscosity (cP) | Water Content | Specific gravity | API gravity | Calorific Value (kkal/kg) |
|------------------------|----------------|---------------|---------------|-----------------|-------------|-------------------------|
| 0                      | 0.998          | 30.192        | 1.040         | 1.040           | 4.475       | 10,241                  |
| 5                      | 0.995          | 29.398        | 1.037         | 1.037           | 4.866       | 10,249                  |
| 6                      | 0.963          | 28.594        | 1.004         | 1.004           | 9.373       | 10,350                  |
| 7                      | 0.997          | 29.740        | 1.039         | 1.039           | 4.596       | 10,243                  |

Based on the test results, it is seen that the greater the percentage of zeolite catalyst used, the smaller the moisture content. This is consistent with the theory that one of the zeolite catalyst properties is the adsorption property [39]. Through pores found in zeolites, zeolites can function as absorbers of gases or liquids. The amount of water molecules absorbed according to the number of pores or the total amount of pore volume of zeolites. Water content in fuel oil is one of the benchmarks of the quality of fuel oil. The smaller the water content in fuel oil, the better the quality. The bio-fuel obtained was then characterized by physical properties such as density, viscosity, specific gravity, API gravity and calorific value and chemical analysis using GC-MS tools to determine the chemical components contained in the bio-fuel.

The value of bio-fuel density without catalyst is greater than the value of bio-fuel density by using zeolite catalyst, as shown in table 1.3. Density of bio-fuel has decreased on catalyst 5% and 6% and has increased on catalyst 7%. The decrease in the value of bio-fuel density in zeolite catalysts of 5% and 6% is due to zeolites having pores that can be utilized as a place in the cracking process, so that the cracking of long-chain hydrocarbons to short-chain hydrocarbons increases with the increasing percentage of zeolite catalysts, which results in molecular weight lower. Whereas in the 7% zeolite catalyst there was an increase in density again, this was due to the use of too much catalyst causing the catalyst to be deactivated by the reactants and abundant products, so that it could cover the active cycle of the catalyst. The use of too much catalyst can also cause molecules that have formed will react back to heavier molecules [38].

Bio-fuel density values obtained are in accordance with the bio-fuel density quality standards for fuels because they are in the range of 0.94-1.11 gr / ml [40]. If the fuel contains many compounds with high molecular weight, it will complicate the evaporation process in the engine combustion chamber, and tends to be a burnt jar that is not completely burned [41]. Mohan [42] revealed that the smaller the density of bio-fuel, the better it will be used as fuel because it is lighter. High density can cause imperfect combustion reactions that can increase emissions and wear on the engine.

The value of bio-fuel viscosity obtained from the pyrolysis of corncobs is in accordance with the quality standard of bio-fuel viscosity for fuel because it is within the range of bio-fuel standards. Dynamotive [43] explains that the viscosity of bio-fuel for fuel is in the range of 10-150 cP. The value of bio-fuel viscosity without catalyst is greater than the average value of bio-fuel viscosity using catalyst, as shown in table 3. Bio-fuel viscosity has decreased in zeolite catalyst 5% and 6% because the catalyst breaks down high molecular weight compounds into hydrocarbon compounds that have low molecular weight and bio-fuel viscosity has increased in catalyst zeolite 7%, the increase in viscosity is due to compounds with high molecular weight. This is consistent with the results of GC-MS analysis which shows the compounds in bio-fuel with 7% zeolite catalyst have a longer retention time which indicates the compounds are included in compounds with high molecular weight [44].

Based on the results of the analysis of specific gravity and API gravity, it can be seen that the results of pyrolysis of corncobs on the 6% catalyst have the lowest specific gravity value and the highest API gravity value. According to Rizky et al. [45], specific gravity with a range of 0.93-1 included in the classification of heavy oil. Heavy oil is a type of oil with high viscosity. This classification based on specific gravity aims to determine the treatment of bio-fuel in the distillation
column if it is going to be purified/extracted thermally and to predict the results of the processed bio-fuel produced.

Based on the analysis of the heating value, the heating value of bio-fuel without catalyst is lower than the heating value of bio-fuel using zeolite catalyst, as shown in Appendix 5. According to Wiratmaja [46] the calorific is inversely proportional to density. Therefore the smaller the bio-fuel density, the higher the calorific value and vice versa, the greater the bio-fuel density, the smaller the calorific value. The calorific value of crude oil generally ranges from 10,160-11,000 kcal / kg, so it can be concluded that the heating value in this study falls within the standard range of calorific value for fuel oil. The calorific value of the fuel is very influential on fuel consumption per unit time. The higher the calorific value of the fuel causes the less fuel consumption.

4. Conclusion

Based on the overall results of the analysis that has been carried out, it can be concluded that the best catalyst concentration is obtained at 6% with the highest volume of 225 ml. The results of the characterization of physical and chemical properties obtained a density of 0.9636 gr / ml, a viscosity of 28.5949 cP and a heating value of 10,349.6602 kcal / kg. The composition of the largest fuel constituent compounds was obtained on 6% zeolite catalyst with a percentage of 56.13% cornhusks (Zea mays L.) waste can be produced as an alternative fuel source as substitute for diesel fuel and conventional fuel.

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