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# Effect of Preparation Method of Palygorskite-Supported Fe and Ni Catalysts on Catalytic Cracking of Biomass Tar

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## Abstract

In this study, the effect of catalyst preparation and additive precursors on the catalytic decomposition of biomass using palygorskite-supported Fe and Ni catalysts was investigated. The catalysts were characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). It is concluded that the most active additive precursor was  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ . As for the catalyst preparation method, co-precipitation had superiority over incipient wetness impregnation at low Fe loadings.

**Keywords:** Biomass tar; Palygorskite; Additive precursor; Catalyst preparation method.

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## 20 **Introduction**

21 Biomass gasification offers the potential for producing a fuel gas that can be used  
22 for power generation systems or synthesis gas applications. Gasification of biomass has  
23 several environmental advantages over fossil fuels, namely lower emission of CO<sub>2</sub> and  
24 other greenhouse gases such as H<sub>2</sub>S, SO<sub>2</sub>, NO<sub>x</sub> [1]. However, one of the major issues in  
25 biomass gasification is dealing efficiently with tar reduction, which presents significant  
26 impediment to biomass gasification systems. The condensable compounds present in tar  
27 may cause problems in downstream equipment, making the cleaning step of biomass  
28 gasification gas necessary in most gasification applications. By far, catalytic  
29 decomposition appears to be a very attractive way to convert tar components into H<sub>2</sub>, CO  
30 and other useful fuels.

31

32 In past several years, most research has focused on steam reforming of various  
33 hydrocarbon feedstock over supported-Ni and precious metal catalysts [2-6]. In general,  
34 Ni catalysts showed high, stable activity for catalytic removal of biomass tar during short  
35 periods. However, coking on the catalyst surface and sintering of Ni particles were  
36 observed and the materials lost their activity under practical conditions over extended  
37 periods of time [7]. Additives play a crucial role in catalyst activity, mainly to improve  
38 catalyst stability. Additives effect the dispersion of active components, resistance to  
39 aggregation of active components, and carbon deposition and sulfur and chlorine

40 poisoning of catalysts [8-11]. To the best of our knowledge, only a few reports on sulfur  
41 or/and chlorine poisoning of catalysts and catalyst preparation exist [12-18]. The  
42 resistance to sulfur poisoning between Ni-WO<sub>3</sub>/MgO-CaO and several commercial  
43 steam-reforming catalysts was examined by Sato *et al.* [16]. The results showed that the  
44 Ni-WO<sub>3</sub>/MgO-CaO catalyst exhibited superior resistance to sulfur poisoning compared  
45 to several commercial steam-reforming catalysts. Therefore, the catalyst needs a high  
46 degree of sulfur or/and chlorine-tolerance because the biomass contains both these  
47 chemicals. These researches are valuable to the development of catalysts for catalytic  
48 cracking of biomass tar. As for catalyst preparation method, the comparison of  
49 Ni/MgO/Al<sub>2</sub>O<sub>3</sub> catalysts prepared by an impregnation-reduction method with the  
50 catalysts prepared by an impregnation-calcination-reduction was investigated by Suo *et*  
51 *al.* [17]. The results revealed that the Ni/MgO/Al<sub>2</sub>O<sub>3</sub> catalyst prepared by the  
52 impregnation-reduction method had larger surface area and smaller particle size. The  
53 effect of Ni catalysts preparation methods on the hydroconversion of the hydrotreated  
54 oil fraction was examined by Masalska [18].

55 Recently, iron-based catalyst and additive Fe attracted attention of researchers.  
56 Nemanova *et al.* [19] reported the use of Fe based catalysts and the effect of several  
57 experimental iron-based granules on biomass tar decomposition. The use of these Fe  
58 based catalysts resulted in the reduction of tar. Liu *et al* [20] investigated the effect of  
59 different additives (Fe, Mg, Mn, Ce) on catalytic cracking of biomass tar over

60 Ni6/palygorskite. The result showed Fe played a better role in improving the reactivity of  
61 Ni6/palygorskite. Based on previous research [20, 21], the effect of preparation method  
62 and additive precursors upon palygorskite-supported Fe and Ni catalysts are reported in  
63 this paper. The objective of this work is to prepare improved catalysts for catalytic  
64 cracking of biomass tar.

65

## 66 **Experimental**

### 67 **Catalysts preparation**

68 Palygorskite used was sampled from Crown Hill, Mingguang city, Anhui province,  
69 China. Its particles size was less than 0.074mm after extrusion, cutting and crushing.  
70 The surface area of the palygorskite was measured as 228.5 m<sup>2</sup>/g. The multi-point BET  
71 surface area of palygorskite was measured using a Beckman Coulter SA-3100 BET  
72 surface area and pore size analyser. The chemical composition measured using a  
73 Shimadzu XRF-1800 with Rh radiation is SiO<sub>2</sub> 65.52 wt%, Al<sub>2</sub>O<sub>3</sub> 5.36 wt%, MgO 13.93  
74 wt%, Fe<sub>2</sub>O<sub>3</sub> 3.19 wt%, FeO 0.42 wt%.

75

76 Palygorskite-supported Ni and Fe catalysts (Fe-Ni/PG) are prepared by incipient  
77 wetness impregnation and co-precipitation. In this study, Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O, FeCl<sub>3</sub> and  
78 FeSO<sub>4</sub> were chosen as the additive precursors. For the catalyst test, all the catalysts were  
79 crushed and sieved to 0.85~0.425 mm, followed by calcination in air for 2 h at 500°C and

80 then reduced in hydrogen at a flow rate of  $80 \text{ ml}\cdot\text{min}^{-1}$ , and held at  $500^\circ\text{C}$  for 1h. The  
81 catalyst sample was identified as  $\text{Fe}_x\text{-Ni}_y/\text{PG}$ , where x denoted the loading of Fe, and y  
82 denotes the loading of Ni in moles. For example,  $\text{Fe}_6\text{-Ni}_6/\text{PG}$  denoted the sample of PG  
83 supported 6 wt%Ni and 6 wt% Fe. Palygorskite-supported Ni and Fe catalysts ( $\text{Fe-Ni}/\text{PG}$ )  
84 were prepared by incipient wetness impregnation. Palygorskite was mixed with aqueous  
85 solutions of  $\text{Ni}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$  and  $\text{Fe}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}$  or  $\text{FeCl}_3$  or  $\text{FeSO}_4$ , followed by drying  
86 at  $105^\circ\text{C}$  overnight, calcined and reduced.

87

88 Palygorskite was impregnated with metal salts by pore volume wetness impregnation.  
89  $\text{Ni}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$  (29.7 g),  $\text{Fe}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}$  (43.3g), and 86 ml of deionized distilled water  
90 were placed in a 150 ml beaker. After dissolution, the mixed liquor was poured into 88 g  
91 PG clay. The mixture was stirred for 20 min. After stirring, the impregnated samples  
92 were aged at room temperature overnight, dried at 378K, cooled to room temperature,  
93 and ground and sieved to obtain particles of 20–40 mesh. For the co-precipitation method,  
94 deionized water is added into beaker with 88g palygorskite. The palygorskite is  
95 suspended by stirring. Then a mixture with  $\text{Ni}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$  (29.7 g) and  $\text{Fe}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}$   
96 (43.3g) were added to the beaker and stirred continuously. After about 10 minutes,  
97 sodium hydroxide was added to make the  $\text{Fe}^{3+}$  and  $\text{Ni}^{2+}$  co-precipitated on the  
98 palygorskite surface by regulating the pH at 8 pH units. The mixture was then dried at  
99 378 K, ground and sieved to obtain particles of 20–40 mesh.

100

## 101 **Catalytic tests**

102       The reaction of the biomass tar, derived from a rice hull gasification power plant  
103 was catalyzed to reduce the tar content and to produce H<sub>2</sub>-rich gas. The tar consists of  
104 79.2 wt% C, 5.3 wt% H, 1.6 wt% N, 7.4 wt% O, 5.3 wt% ash and a small amount of Cl  
105 and S elements.

106

107       Catalytic tests were controlled by using a fixed-bed experimental system shown in  
108 Fig. 1. The setup consisted of three kinds of systems (a sample injection system including  
109 biomass tar and carrier gas, a reaction and collection system and an analysis system). The  
110 reactor used was a straight quartz tube operating at 700°C (id. 30 mm width and 400 mm  
111 body length) and the catalyst bed was supported by means of quartz wool. All the  
112 catalysts were tested under the same experimental conditions: WHSV = 2.7 h<sup>-1</sup> and  
113 atmospheric pressure. 10 g of catalyst was used in each experiment. Nitrogen was used to  
114 carry the biomass tar cracking gases into and out of the reactor; a flow rate of nitrogen  
115 was 80 ml·min<sup>-1</sup>. The rice hull gasification tar was introduced into the reactor with a  
116 peristaltic pump (BQ50-1J) at a flow rate of 0.45 g·min<sup>-1</sup>. Tar was introduced as a liquid  
117 in a silica tube. The tar did not stick to reaction walls because a special set up was used  
118 during the experiment. The tar collected from rice hull gasification power plant is thick.  
119 Thus, the introduction lines were not heated. Before and after the reaction, the

120 introduction lines were weighed to calculate the mass of tar in the reactor. The duration  
121 of the experiment was 20 mins. Granular activated carbon (GAC) was used for  
122 collecting unreacted tar and by-products as shown in Fig. 1.

123

124 Almost all the unreacted and by-products can be absorbed by GAC. In this way,  
125 unreacted tar and byproducts were recovered in GAC and the amounts of unreacted tar  
126 and byproducts were calculated by subtracting the mass of the filter before reaction from  
127 that remaining after reaction. Thus, the efficiency of catalysts on biomass tar can be  
128 calculated using the following formula. In the formula,  $M_{in-tar}$  and  $M_{out-tar}$  represent the  
129 amount of tar dragged into the reactor and tar collected in the filter, respectively.

130 Tar conversion =  $\frac{M_{in-tar} - M_{out-tar}}{M_{in-tar}} \times 100\%$

131

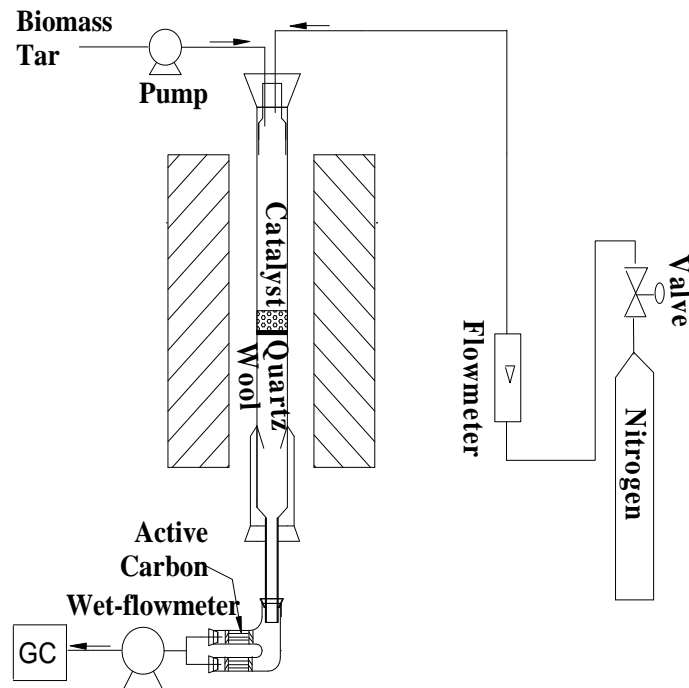
132 After the reaction, the peristaltic pump was switched off and resultant gases were  
133 carried by nitrogen into an air cell and the gas volume was determined by a wet test  
134 meter. The non-condensable gases, which included hydrogen, nitrogen, carbon monoxide  
135 and methane were analyzed by gas chromatography (GC-7890T) equipped with a C2000  
136 column and a thermal conductivity detector (TCD) with argon as carrier gas to measure  
137  $H_2$ ,  $N_2$ ,  $CO$  and  $CH_4$ . The column temperature, evaporation chamber temperature, the  
138 temperature and bridge current of detector were  $70^\circ C$ ,  $120^\circ C$ ,  $100^\circ C$ , 100 mA,



139 respectively. Hydrogen yield was calculated by the following formula.  $M_{H-out}$  and  $M_{H-in}$   
 140 represent the mass of hydrogen derived from catalytic cracking of tar and the mass of  
 141 hydrogen element in tar dragged into the reactor, respectively.

142 Hydrogen yield =  $\frac{M_{H-out}}{M_{H-in}} \times 100 \%$

143



144

145

146

**Fig. 1. The system for activity measurement.**

147

148 **Catalyst characteristic**

149 X-Ray diffraction (XRD) measurements were performed using a Rigaku powder  
 150 diffractometer with Cu  $K\alpha$  radiation. The tube voltage was 40 KV and the current was  
 151 100 mA. The XRD diffraction patterns were taken in the range of 5-70° at a scan speed

152 of  $4^\circ \text{ min}^{-1}$ , which was used for identifying Ni and Fe as well as their oxidation states on  
153 the surface of palygorskite. Phase identification was carried out by comparison with the  
154 database cards.

155

156 Transmission electron microscopy (TEM) were made by a JEOL 2010 microscope.  
157 The samples were mixed with alcohol and deposited on a Cu grid covered with a  
158 perforated carbon membrane.

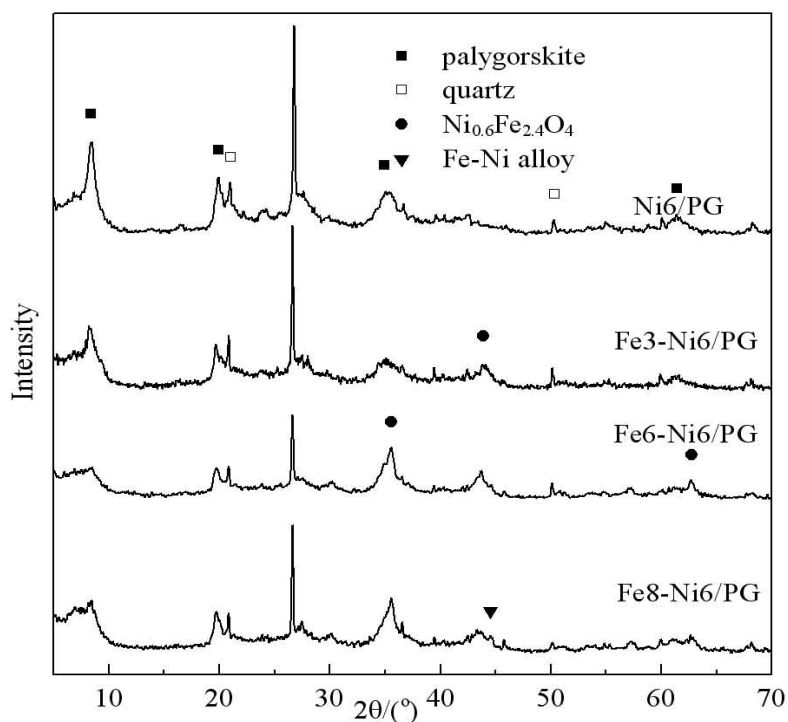
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### 160 **3. Results and Discussion**

#### 161 **3.1 XRD result**

162 Fig. 2 shows the XRD patterns of Ni6/PG, Fe3-Ni6/PG, Fe6-Ni6/PG, Fe8-Ni6/PG  
163 catalysts prepared with incipient wetness impregnation. Four major peaks at  $8.4^\circ$ ,  $19.7^\circ$ ,  
164  $35.1^\circ$ ,  $61.4^\circ$  were indentified as PG. The diffraction peaks corresponding to PG become  
165 more intense with the decreasing Fe loading. Additionally, the peaks at  $35.5^\circ$ ,  $43.7^\circ$ ,  
166  $62.6^\circ$  were observed and these were identified as  $\text{Ni}_{0.6}\text{Fe}_{2.4}\text{O}_4$ . The intensities of these  
167 peaks assigned to  $\text{Ni}_{0.6}\text{Fe}_{2.4}\text{O}_4$  increased with an increase in the Fe loading. Furthermore,  
168 the peak at  $44.6^\circ$ , assigned to the alloy of Fe and Ni was confirmed by XRD. Fig. 3  
169 displays the XRD patterns of Ni6/PG, Fe3-Ni6/PG, Fe6-Ni6/PG, Fe8-Ni6/PG prepared by  
170 the co-precipitation method after reduction. Five major peaks at  $8.4^\circ$ ,  $19.8^\circ$ ,  $34.3^\circ$ ,  $35.2^\circ$   
171 and  $42.5^\circ$  for  $2\theta$  are identified as palygorskite and it is found that the intensity of these

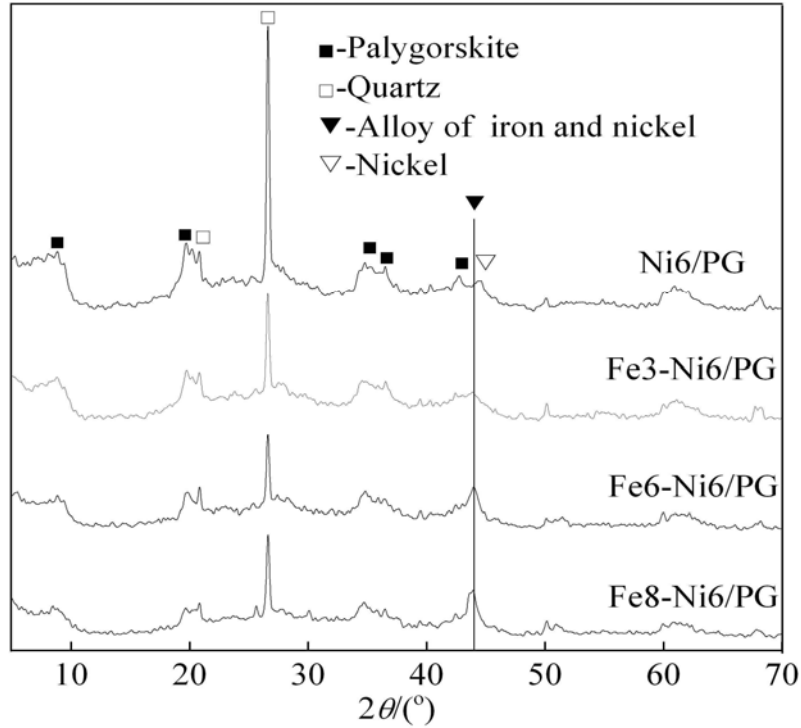
172 peaks increase with the increasing Fe content. It is also found that the peaks at 21.4° and  
 173 27.5° assigned to SiO<sub>2</sub> become less intense with the increasing Fe loading. Additionally,  
 174 the peak at 44.7° for 2θ is identified as the Ni metal phase on Ni6/PG catalyst formed by  
 175 the reduction of NiO by hydrogen. Instead of the peak, the peak at 44° assigned to the  
 176 alloy of Fe and Ni is observed over the Fe-Ni6/PG catalyst and the intensity of the peak  
 177 increased with the increasing Fe content. However, the Ni metal phase is not found on  
 178 the Ni6/PG catalyst prepared with incipient wetness impregnation. The spinels of Fe and  
 179 Ni were not found in Fe-Ni6/PG prepared by co-precipitation. The more highly active  
 180 components are dispersed, the easier these metal elements were reduced. That is to say,  
 181 co-precipitation can improve the dispersion of active component on support, which  
 182 reduces the reduction temperature of nickel oxide and iron oxide.



183

184  
185

**Fig. 2. XRD patterns of Fe-Ni6/PG prepared with incipient wetness impregnation.**



186  
187  
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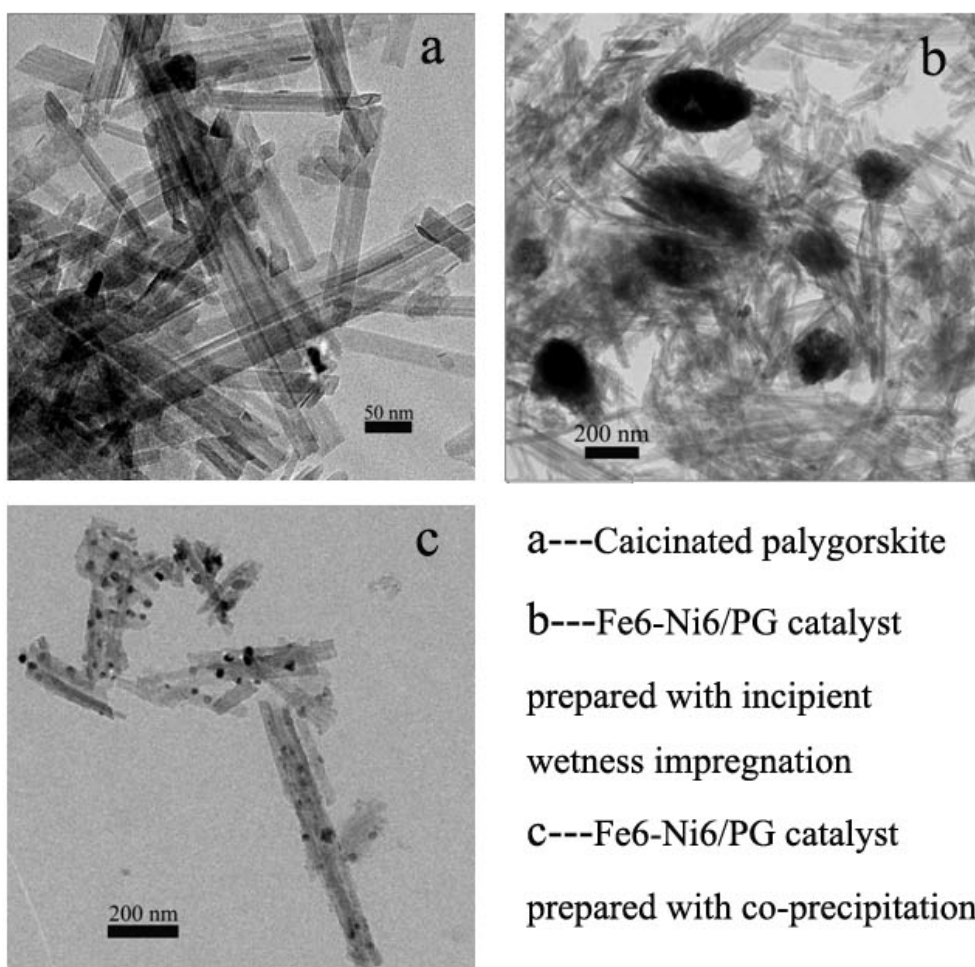
**Fig. 3. XRD patterns of Fe-Ni6/PG prepared with co-precipitation.**

### 189 3.2 TEM results

190 The TEM images of palygorskite calcinated at 500 °C and Fe6-Ni6/PG prepared  
191 with incipient wetness impregnation and co-precipitation are presented in Fig. 4. The  
192 images indicate that some particles are observed on the Fe6-Ni6/PG catalyst compared to  
193 palygorskite. This is in good agreement with the corresponding XRD patterns. XRD  
194 patterns of Fe6-Ni6/PG prepared with incipient wetness impregnation and  
195 co-precipitation show the existence of alloy and/or spinel of Ni and Fe. However, as  
196 shown in Fig. 4 (b, c), some larger particles (100-400 nm) are found on the support  
197 prepared with incipient wetness impregnation than these (5-40 nm) on palygorskite

198 prepared with co-precipitation. Some highly dispersed nanoparticles are observed on the  
199 palygorskite in Fig 4 (c). The dispersion of catalysts prepared with co-precipitation  
200 appears superior to that of catalysts prepared with incipient wetness impregnation. In  
201 addition, the previous study [20] proved that the particles on palygorskite becomes larger  
202 and larger with the increasing Fe loading.

203



204

205

**Fig. 4. TEM of PG and Fe8-Ni6/PG catalyst.**

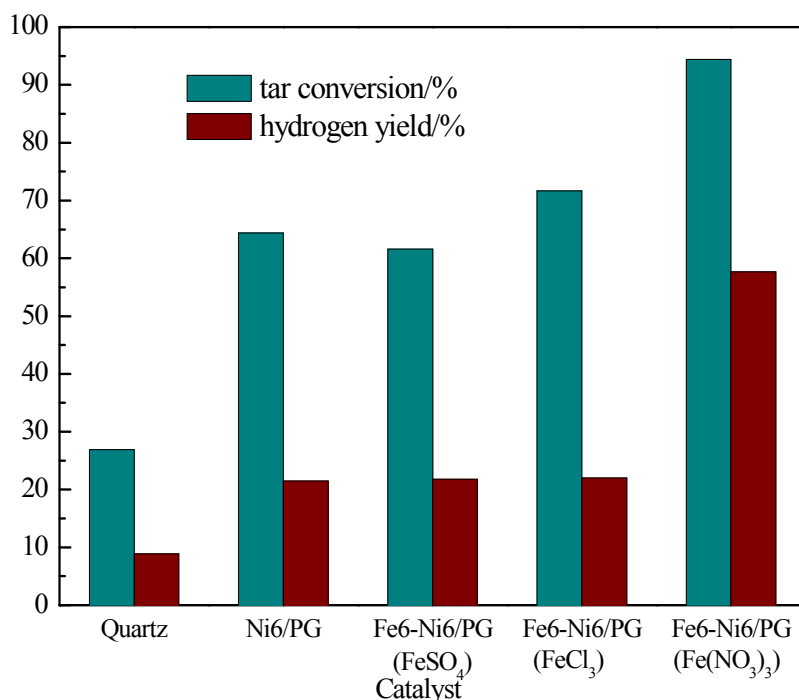
206

### 207 **3.3 Effect of Additives Precursor on Fe6-Ni6/PG Catalyst**

208

209 It was mentioned earlier that in this reactor system, Fe plays an important role in  
210 catalytic cracking of biomass tar using Ni6/PG catalysts [20]. Fig. 5 shows the tar  
211 conversion and H<sub>2</sub> yield obtained from the catalytic decomposition of biomass tar with  
212 the Fe6-Ni6/PG catalyst as a function of an Fe additive precursor. It is evident the tar  
213 conversion and H<sub>2</sub> yield increased in the presence of Fe6-Ni6/PG and Ni6/PG catalysts  
214 compared with a quartz catalyst. On the other hand, the Fe additive precursor influences  
215 the increase in tar conversion and H<sub>2</sub> yield. In the case of the Ni6/PG catalyst modified  
216 by Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O, it is found that tar conversion and H<sub>2</sub> yield obtained the highest  
217 values, i.e. 94.4% and 57.7%. However, the decrease in tar conversion and H<sub>2</sub> yield  
218 obtained over the Ni6/PG catalyst modified with FeSO<sub>4</sub> or FeCl<sub>3</sub>, indicates that sulfur or  
219 chlorine poisoning occurs over the Ni6/PG catalyst modified by FeSO<sub>4</sub> or FeCl<sub>3</sub>. The  
220 addition of FeSO<sub>4</sub> has a negligible or even negative effect. This is good agreement with  
221 theoretical research [22]. Ni catalysts can be deactivated by sulfur containing compounds.  
222 In conclusion, the order of activity over Ni6/PG modified by different Fe precursors is as  
223 follows: Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O > FeCl<sub>3</sub> > FeSO<sub>4</sub>.

224



225

226 **Fig. 5. Effect of additive precursor on catalytic cracking of biomass tar over an Fe6-Ni6/PG**  
 227 **catalyst.**

228

### 229 **3.4 Effect of preparation method of Fe6-Ni6/PG catalyst**

230 The occurrence of a synergic effect between nickel oxide and iron oxide has directed  
 231 our attention to the problem of how the method of Fe6-Ni6/PG catalyst preparation  
 232 influenced the catalyst activity. In the experiment, the catalyst was prepared by incipient  
 233 wetness impregnation and co-precipitation. From the study of the effect of an additive  
 234 precursor on the decomposition of tar and hydrogen yield, it is suggested that additive  
 235 precursor plays a crucial role in tar conversion and hydrogen yield. Therefore,  
 236 Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O is selected as the additive precursor to investigate the effect of catalyst

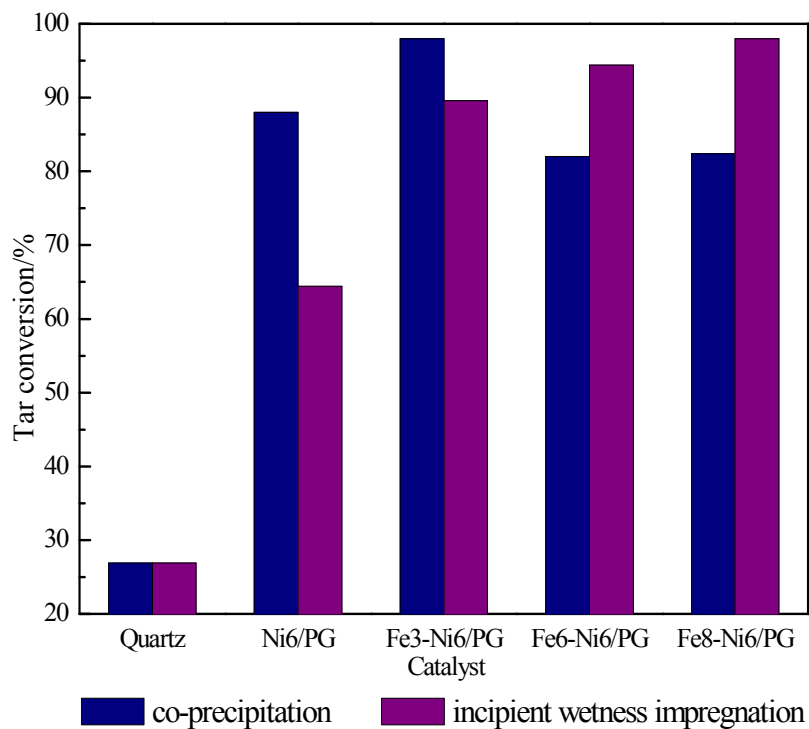
237 preparation methods.

238

239 The effect of catalyst preparation on tar conversion is depicted in Fig. 6. As shown  
240 in Fig. 6, these two catalyst preparations presented obvious increase in tar conversion  
241 compared to the decomposition of tar over quartz. From the comparison between the  
242 incipient wetness impregnation and co-precipitation, it is found that tar conversion  
243 increases firstly and then decreases with an increasing additive loading for  
244 co-precipitation and comes to the highest value (98%) when Fe content is 3%. With  
245 respect to incipient wetness impregnation tar conversion increased with an increasing  
246 additive loading and has a highest tar conversion (98%) when the Fe content is 8%. That  
247 is to say, co-precipitation methodology shows superiority over incipient wetness  
248 impregnation at a low loading of the additive. The effect of catalyst preparation on  
249 hydrogen yield is presented in Fig. 7. It is found that the hydrogen yield over different  
250 catalysts is similar with tar conversion. The highest value of hydrogen yield is 56.5% and  
251 58.5% for co-precipitation and incipient wetness impregnation, respectively. Namely,  
252 hydrogen yield over Fe<sub>3</sub>-Ni<sub>6</sub>/PG prepared with co-precipitation is higher 25.7% than  
253 incipient wetness impregnation, attributed to the high dispersion of the active component  
254 on palygorskite.

255

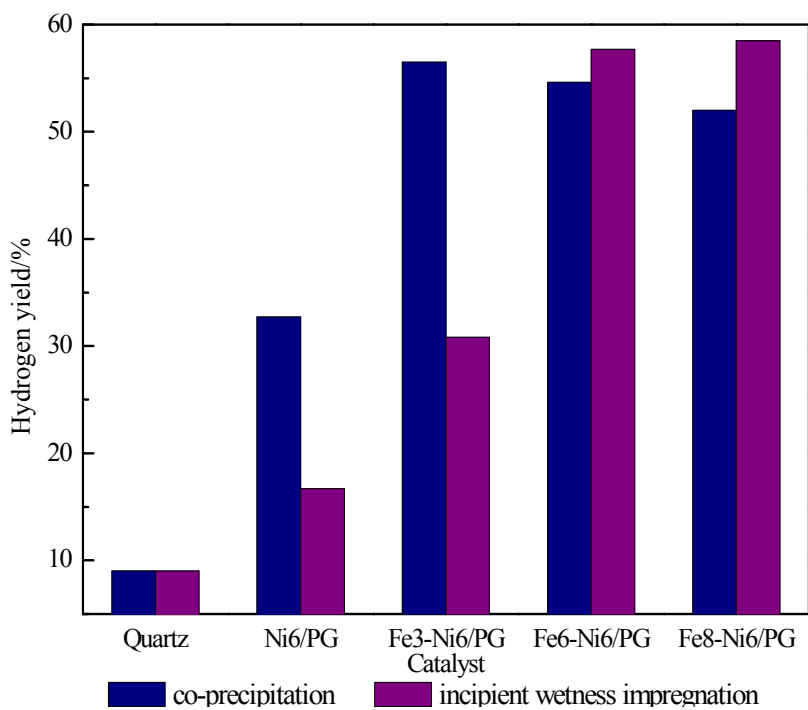




256

257

**Fig. 6. The effect of catalyst preparation on tar conversion.**



258

259

**Fig. 7. The effect of catalyst preparation on hydrogen yield.**

260

## 261 **Conclusions**

262 The results of characterization (XRD and TEM) for a Ni6/PG catalyst prepared with  
263 co-preparation shows that Ni metal particles are formed over the catalyst. A few particles  
264 of an alloy of Fe and Ni were obtained over the Fe-Ni6/PG catalyst prepared with  
265 co-precipitation.

266

267 As for the catalytic performance, it is found that the Ni6/PG catalyst modified by  
268  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  showed a higher catalytic performance for tar conversion (94.4% tar  
269 conversion, 57.7% hydrogen yield). In the case of the catalyst preparation, it can be  
270 concluded that co-precipitation shows a higher activity over Fe-Ni6/PG at low Fe  
271 contents (<3%) due to the high dispersion of active component on PG. The mechanism of  
272 catalytic decomposition of biomass tar over palygorskite- supported Fe and Ni will be  
273 investigated in a following study.

274

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278

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