



Attrition of methanol to olefins catalyst in jet cup



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ARTICLE INFO

Article history:

Received 16 May 2016

Received in revised form 24 December 2016

Accepted 30 December 2016

Available online 2 January 2017

Keywords:

Attrition

MTO

Catalyst

Jet cup

Gwyn formulation

ABSTRACT

Attrition of catalyst in a fluidized bed reactor is an inevitable issue especially in a commercial unit. Methanol to olefins (MTO) is becoming one of the main stream technologies for light olefins production. The attrition of MTO catalyst, however, received little attention. This study is focused on the attrition behavior of MTO catalyst in jet cup at high temperature. The influence of test time, inlet gas velocity, and temperature on MTO catalyst attrition was studied. It is found that the Gwyn formulation can well represent the relation between attrition index and test time. Our results show that jet cup can retrieve results quantitatively comparable to high velocity gas jets method while significantly shortening test time. It is also found that the inlet gas velocity has considerable influence on the MTO catalyst attrition, and the relation between inlet gas velocities and attrition index can be described by a power index of 3.7. Similar to high velocity gas jets experiments the attrition index manifests a maximum with the increase of temperature. But the temperature corresponding to the maximum attrition index shifts from 300 °C in high velocity jets tests to 100 °C in jet cup experiments. An analysis based on SEM pictures indicates that the transition of attrition mechanism is responsible for this shift. An empirical correlation has been presented for MTO catalyst attrition in jet cup, which shows good agreement with experimental data for inlet gas velocity from 88 to 158 m/s, and temperature from 100 to 500 °C.

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1. Introduction

Fluidized bed is one of the widely used reactors in chemical industry due to its excellent performance such as perfect fluid–solid contact, efficient heat and mass transfer between fluid and solid, uniform temperature distribution and good thermal stability in the reactors and so on. However, particles attrition and the wear of inner walls of the equipment are the highly concerned disadvantages especially for the pilot and commercial plants. Continuous movement of particles inside fluidized bed will cause frequent particle–particle and particle–wall collisions, which, in many applications, can result in severe decrease in particles' strength and eventually lead to surface abrasion and/or breakage of particles, wear of the equipment as well. Therefore, research on catalyst attrition in fluidized beds is of practical significance in catalytic process development. Methanol to olefins (MTO) is a recently developed catalytic process for on-purpose ethylene and propylene production with methanol as feedstock. MTO starting from coal was first commercialized in 2010 in China by Dalian Institute of Chemical Physics, Chinese Academy of Sciences. MTO is becoming one of the main stream technologies for light olefins production. In MTO process, fluidized beds were used as reactor and regenerator, sharing similarity

with the fluid catalytic cracking (FCC) process in refineries. In the past decades, the attrition of FCC catalyst was a subject of intensive study [1–11]. The attrition of MTO catalyst, however, received little attention.

Typically catalyst attrition in fluidized bed reactors can be due to complicated mechanical, thermal, and chemical stress experienced by catalyst particles. Thus both material properties and operation conditions will influence the attrition rate of catalyst in fluidized beds. One of the challenges in catalyst attrition study is that the attrition process of catalyst in an industrial fluidized bed reactor can span months or even years. In laboratory the research with such a time scale, if not impossible, is unrealistic. In this regards, a variety of research methods [12] have been developed to study catalyst attrition in laboratory. For example, single particle impact experiments [13], shear tests [14], drum tests [15], high velocity gas jets [10,11,16] and jet cup [9,17,18]. Amongst these methods, high velocity gas jets and jet cup are commonly-used for bulk attrition testing of fluidized bed catalyst [9,19]. But there are certain differences between these two methods. In high velocity gas jets method, high speed gas flow passes through one or three holes of micrometer size in a distribution plate, agitating severe collisions between particles. In the jet cup method, gas enters the vessel tangentially. In this case particles will be dragged and move upwards spirally, experiencing frequent collisions with the inner wall of the jet cup. Weeks and Dumbill [9] and Zhao et al. [19] compared these two methods, and found that these two test methods are comparable in terms of attrition propensity ranking. But jet cup method requires

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fewer samples and can speed catalyst attrition-resistance determination. The early type of jet cup was cylindrical with small diameter, which had been improved afterwards by Cocco et al. [18]. Jet cup method has been used effectively to assess the attrition resistance of FCC catalyst [1,2,9], slurry bubble column reactor catalysts [20], spray-dried iron Fischer-Tropsch catalysts [21–24], oxygen carrier particles for chemical looping combustion [25,26] and so on.

In a previous study [27,28] we studied the attrition of commercial MTO catalyst by use of a high velocity air jet unit. It has been discovered that the attrition mechanism of MTO catalyst at room temperature is different from that at high temperature. Thus catalyst attrition resistance evaluation at room temperature might not reflect the situation at high temperature. In addition to that, it is suggested that at least 24 h is required for an attrition test in high velocity gas jets facility to achieve an equilibrium state for MTO catalyst. The purpose of the current study is to investigate the attrition of MTO catalyst in a jet cup at high temperature. Although the jet cup method has been used extensively, most of work was carried out at room temperature. We will focus on the comparison of our jet cup results with those obtained via high velocity gas jets, and try to establish an empirical correlation of MTO catalyst attrition index with jet cup operation parameters such as inlet gas velocity, temperature and test time.

2. Experimental

2.1. Jet cup attrition apparatus

Fig. 1 shows the jet cup experimental unit used in this work. Cocco et al. [18] studied particle motion in five different types of jet cups by use of cold flow experiments and CFD simulations. They found that the conical jet cup is more suitable for quantitative test of particle attrition, and testing results could be readily related to the attrition loss rates in fluidized bed cyclones. Therefore a conical jet cup made of stainless steel was adopted in our work. The jet cup unit can be heated up to 600 °C by a furnace. The top part of the settling chamber that was not enclosed by

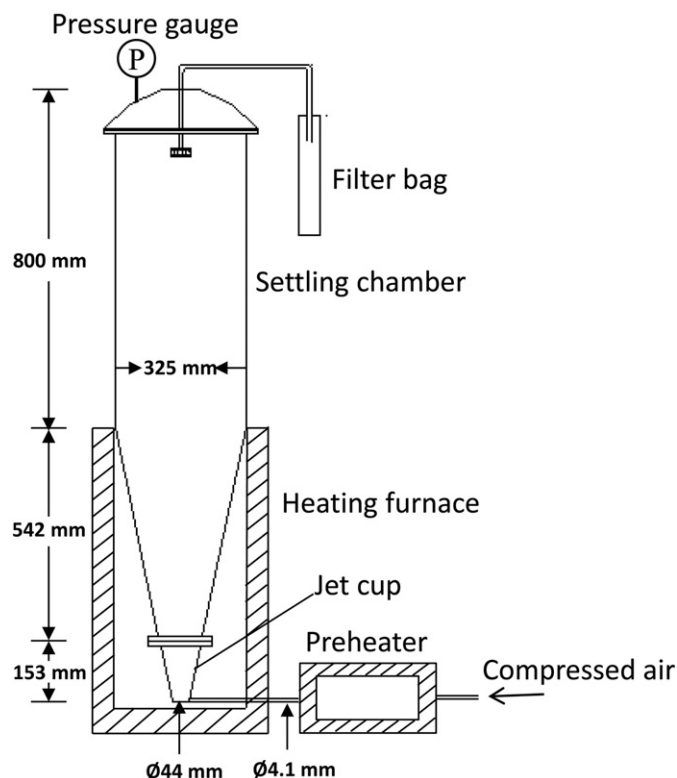


Fig. 1. Experimental apparatus.

the furnace was covered with thermal isolation cotton. A preheater was installed to heat the compressed air the required temperature before entering the jet cup, which minimizes the effect of cool gas on catalyst attrition.

The conical jet cup is 153 mm high, with diameter of 44 mm at bottom and 73 mm at top. A small tube of diameter of 4.1 mm is tangentially connected to the bottom of jet cup, serving as the gas inlet. The jet cup is attached to the settling chamber by flange connection. The settling chamber is cylindrical at the top and conical at the bottom. The diameter and height of the cylindrical part are 325 and 800 mm, respectively. The conical part is 542 mm high, with a small diameter of 86 mm at the inlet and a large diameter of 325 mm at the outlet.

2.2. Catalyst materials

Fresh commercial MTO catalyst was used in this work. Prior to each test, the catalyst sample was sieved to remove fines. The particle size distribution (PSD) of the sample is shown in Fig. 2. The loose bulk density is 0.75 g/cm³, the median particle diameter (d_{p50}) and Sauter mean diameter (d_{p32}) are about 112 μm and 107 μm respectively.

2.3. Attrition measurements

The sieved catalyst sample was heated in a muffle furnace for 3 h at 600 °C and cooled down to room temperature in a vacuum desiccator. Then 100 g powder was weighed and charged into the jet cup. After the flange connection between the jet cup and settling chamber had been tightened, the gas flow started. Before the gas flow meter was adjusted to the desired value, a gas leakage check was carried out. Each test lasted for 3 h. There are three streams of catalyst particles that need to be collected after each test. The first stream is fine particles entrained from the outlet of settling chamber, which were collected by filter bags. The second and third stream are catalyst remaining in the jet cup and fines adhering to the inner wall of the unit. After each test, all three streams of catalyst were weighed and analyzed. A material balance analysis showed that the maximum fine loss was approximately 1.5% of the initial sample for all tests.

PSD of catalyst was analyzed by Malvern laser particle size analyzer (Mastersizer 3000). A total PSD of the sample after each test was calculated based on PSD of each stream of catalyst particles according to the weight fraction. The details were described in our previous publication [27]. The morphology of catalyst was observed by a scanning electron microscope (SEM, Hitachi™ 3000). In this work, attrition index (*AI*) is used to characterize the attrition of MTO catalyst. *AI* is defined as the weight percentage of particles smaller than 20 or 44 μm (expressed as AI_{20} and AI_{44} , respectively) in a sample after a certain duration of test.

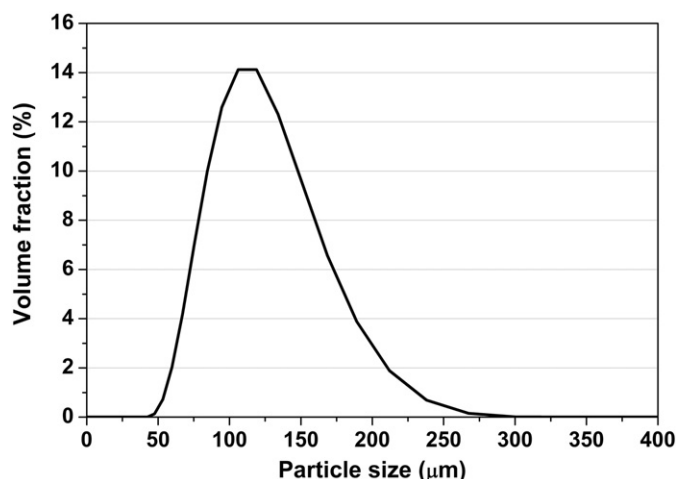


Fig. 2. Particle size distribution of the fresh catalyst sample.

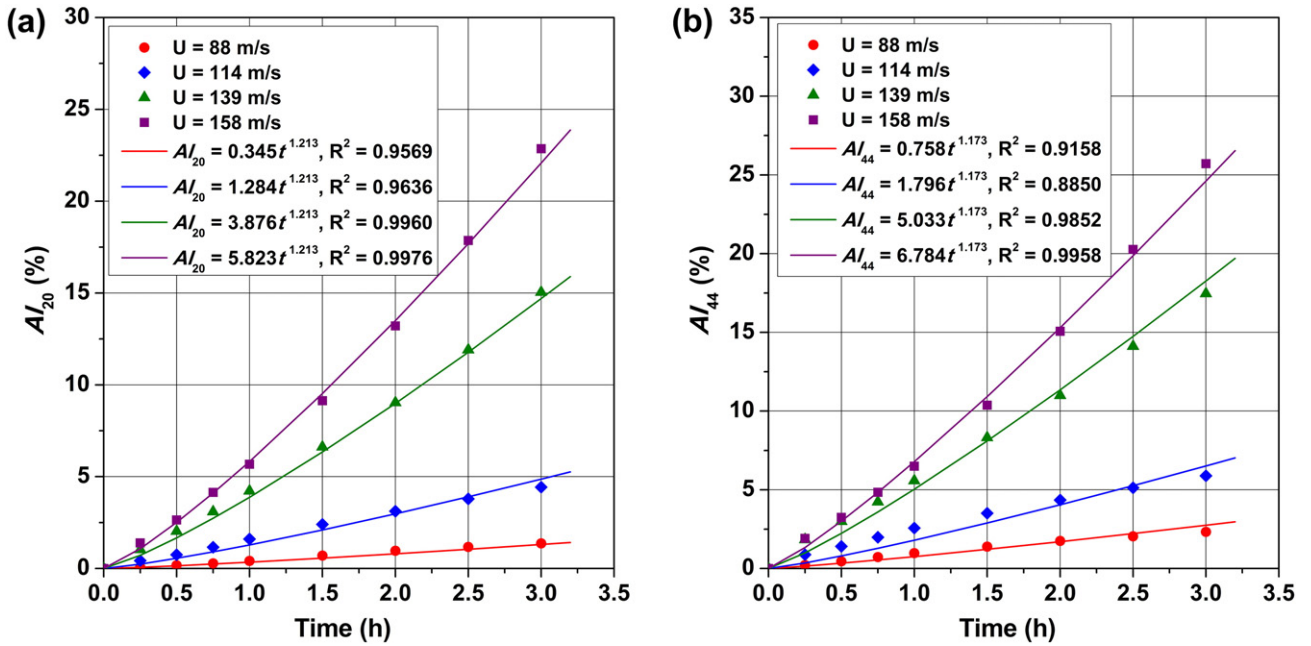


Fig. 3. AI as a function of test time for different inlet gas velocities (88, 114, 139, and 158 m/s) at fixed temperature of 100 °C: (a) AI₂₀, (b) AI₄₄.

3. Results and discussion

3.1. The influence of test time

We first examined the influence of test time on attrition index (AI) in jet cup. Four inlet gas velocities ($U = 88, 114, 139,$ and 158 m/s) and five temperatures ($T = 100, 200, 300, 400, 500$ °C) were considered. In order to measure AI at different time, we replaced the filter bags attached to the settling chamber in a regular interval i.e. every 15 min in the first hour and every 30 min in the rest. Fines collected by the filter bags were weighed and stored in a sample sack for further off-line PSD and morphology analysis. Fines adhering to the inner wall as well as those remaining in the jet cup cannot be weighted during the course of test. Thus we collected these fines (the second and third stream) only at

the end of test. Fines from the second and third stream were uniformly assigned over the whole test time when calculating the AI. In fact, in our experiments fines from the second and third stream only had a minor impact on the results of AI as most of fines were collected by the filter bags.

In Figs. 3 and 4 typical results of AI are depicted as a function of time. As can be seen, both AI₂₀ and AI₄₄ increase with test time. In our previous study [27], we found that the AI in high velocity air jets with attrition time could be well fitted by Gwyn formulation [11]:

$$AI = k_1 t^n \tag{1}$$

where AI is the weight percentage of particles smaller than 20 or 44 μm (expressed as AI₂₀ and AI₄₄, respectively) in a sample after a certain test

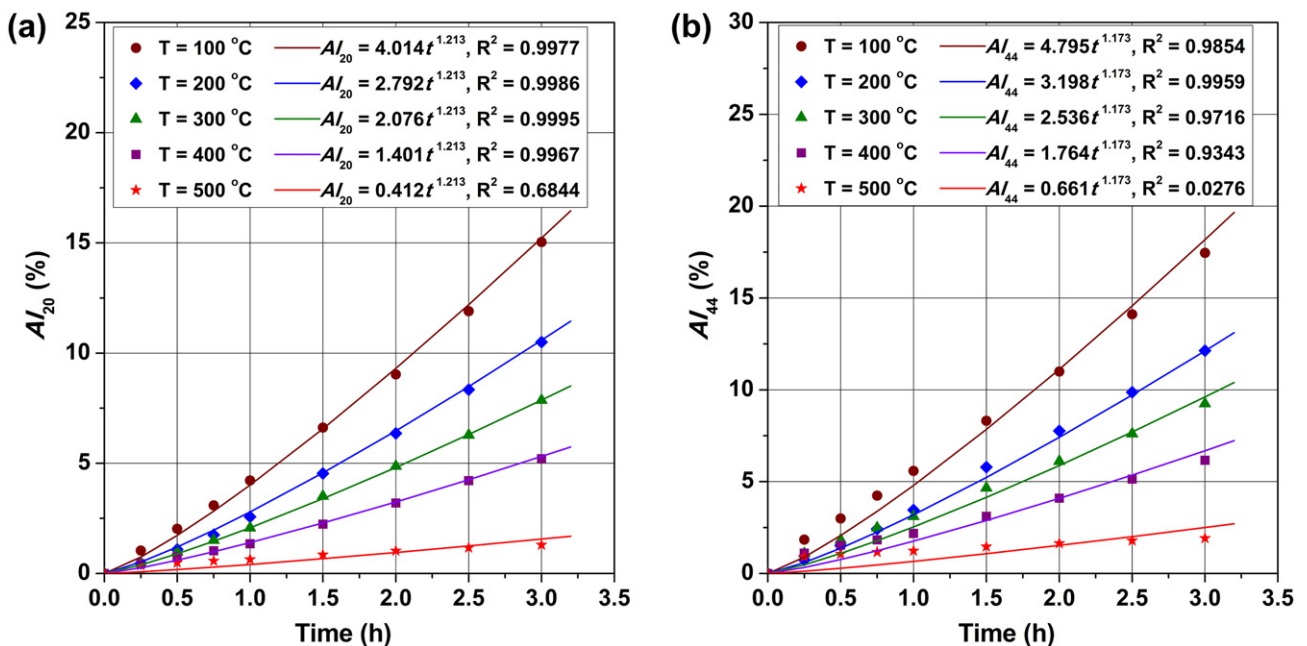


Fig. 4. AI as a function of test time for different temperature (100, 200, 300, 400, and 500 °C) at fixed inlet gas velocity of 139 m/s: (a) AI₂₀, (b) AI₄₄.

Table 1
The parameters k_1 and n for tests with different inlet gas velocities (temperature is 100 °C).

Inlet gas velocity, m/s	Al_{20} k_1, h^{-1}	n	Al_{44} k_1, h^{-1}	n
88	0.345	1.213	0.758	1.173
114	1.284	1.213	1.796	1.173
139	3.876	1.213	5.033	1.173
158	5.823	1.213	6.784	1.173

time, k_1 is attrition rate constant, n is a fitting parameter, and t is test time. In the Gwyn formulation $W = K_p t^n$, where W is the weight fraction of particles less than defined particle size, the constant K_p is a function of initial particles size, and the empirical exponent n is approximately constant for all the catalyst particle sizes in Gwyn's original study and independent of initial particle size. Neil and Bridgwater [14] found the Gwyn formulation represent the attrition occurring in a fluidized bed, a screw pug mill and an annular shear cell very well, and they showed that the parameter n differs little for the same material and is independent of the mechanical equipment used. Klett et al. [29] also observed n was constant for the same catalyst. In our study, for all cases, a linear relationship between $\ln(AI)$ and $\ln t$ could be identified, and the slopes of the linear functions (i.e. the exponent in Gwyn formulation) are almost the same. So, we suppose the parameter n in Eq. (1) is a constant relevant to the material property of catalyst, and independent of the test equipment and operating conditions; k_1 is an empirical parameter related to the equipment and test conditions such as gas velocity, temperature, and so on. Our previous results showed that n obtained from two separate tests in high velocity gas jets matches very well. In current work, we still use Gwyn formulation to fit the AI measured in jet cup. The results are shown in Figs. 3 and 4, and the corresponding fitting parameters k_1 and n are included in Tables 1 and 2. From Figs. 3 and 4, it can be inferred that Gwyn formulation is also suitable for AI of MTO catalyst in jet cup regardless of temperature and inlet gas velocity.

We compared the parameter n obtained in current study with that in high velocity gas jets. In our previous study [27] with high velocity gas jets method, we derived $n = 1.233$ for Al_{20} at room temperature and $n = 1.236$ for Al_{20} at 500 °C. In the jet cup experiments, as shown in Tables 1 and 2, we got $n = 1.213$ and 1.173 for Al_{20} and Al_{44} , respectively. The deviation of Al_{20} is within 2.0% regardless of temperature and attrition test methods. Weeks and Dumbill [9] and Zhao et al. [19] compared the attrition of different types of catalyst and proposed that jet cup and high velocity gas jets are comparable in terms of attrition propensity ranking. The surprising consistence of n for Al_{20} in our work may further suggest that for MTO catalyst the attrition indices measured in both jet cup and high velocity gas jets method are quantitatively comparable. In high velocity gas jets methods, according to our previous study, a relatively long test time is required [27]. It was argued that at least 24 h is necessary in order to achieve an equilibrium attrition rate for MTO catalyst at room temperature, and at an elevated temperature of 500 °C the test time can be reduced to 2 h. In jet cup method, as shown in Figs. 3 and 4, it is apparently shown that an equilibrium attrition rate for MTO catalyst can be obtained after 15 min. This means for MTO catalyst jet cup method can retrieve quantitatively comparable AI as in high velocity gas jets method while significantly shorten test time.

Table 2
The parameters k_1 and n for tests at different temperatures (inlet gas velocity is 139 m/s).

Temperature, °C	Al_{20} k_1, h^{-1}	n	Al_{44} k_1, h^{-1}	n
100 ^a	4.014	1.213	4.795	1.173
200	2.792	1.213	3.198	1.173
300	2.076	1.213	2.536	1.173
400	1.401	1.213	1.764	1.173
500	0.412	1.213	0.661	1.173

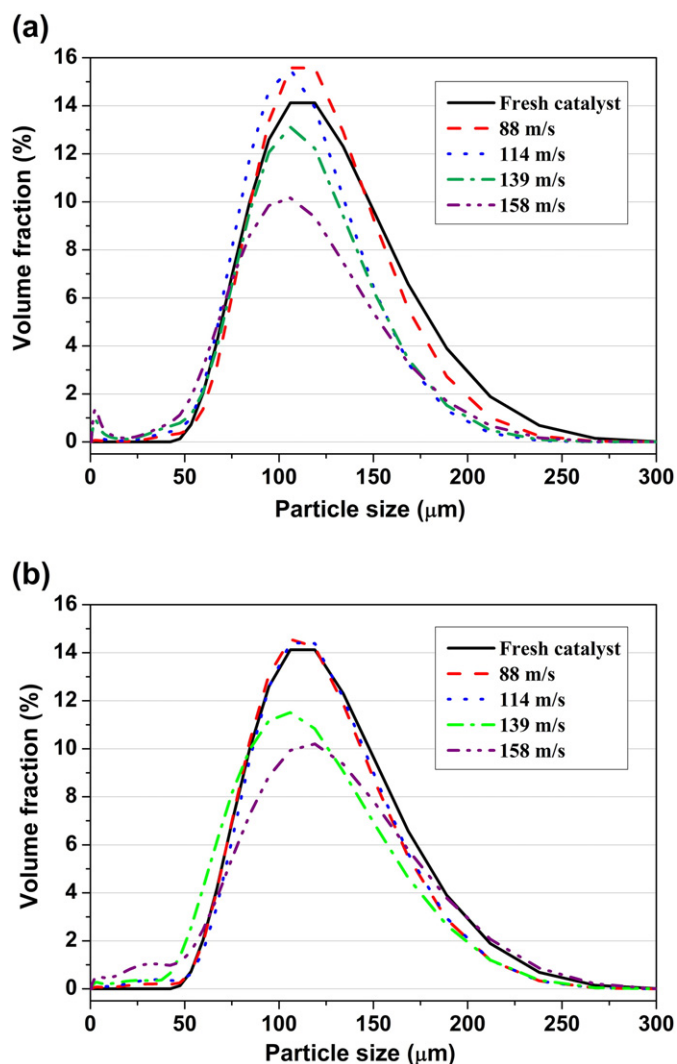


Fig. 5. Particle size distribution of the samples after tests at various inlet gas velocities: (a) 100 °C, (b) room temperature.

3.2. Influence of inlet gas velocity

From Fig. 3, we can find that the inlet gas velocity in jet cup has an considerable impact on MTO catalyst attrition. Fig. 5 shows the total PSD of catalyst samples after tests. A close check with total PSD of catalyst after tests indicates that abrasion is the dominant attrition mode for gas velocity in the range of 88–158 m/s. At 100 °C (Fig. 5(a)) the attrition mode is abrasion, which is evidenced by that there is only one primary peak for the total PSD of catalyst after tests. The peak of PSD shifts from 125 to around 100 μm when the inlet gas velocity increases from 88 to 158 m/s, which suggests that higher gas velocity will enhance the severity of abrasion in jet cup. But at room temperature, a higher gas velocity (158 m/s) can lead to a few fragments in addition to the abrasion, which can be evidenced by the second peak at 30 μm for inlet gas velocity of 158 m/s.

Fig. 6 shows the SEM pictures of catalyst remaining in the jet cup after tests at room temperature, wherein Fig. 6(a) is the initial sample of fresh catalyst particles. Apparent bulges on the surface can be observed for the initial sample, which were probably formed during the catalyst preparation process. These bulges disappear after attrition tests, as shown in Fig. 6(b) to (e). For a relatively low gas velocity (88 to 114 m/s), the sphericity of particles remains intact, and there are almost no fragments, which indicates the attrition mode is abrasion. As

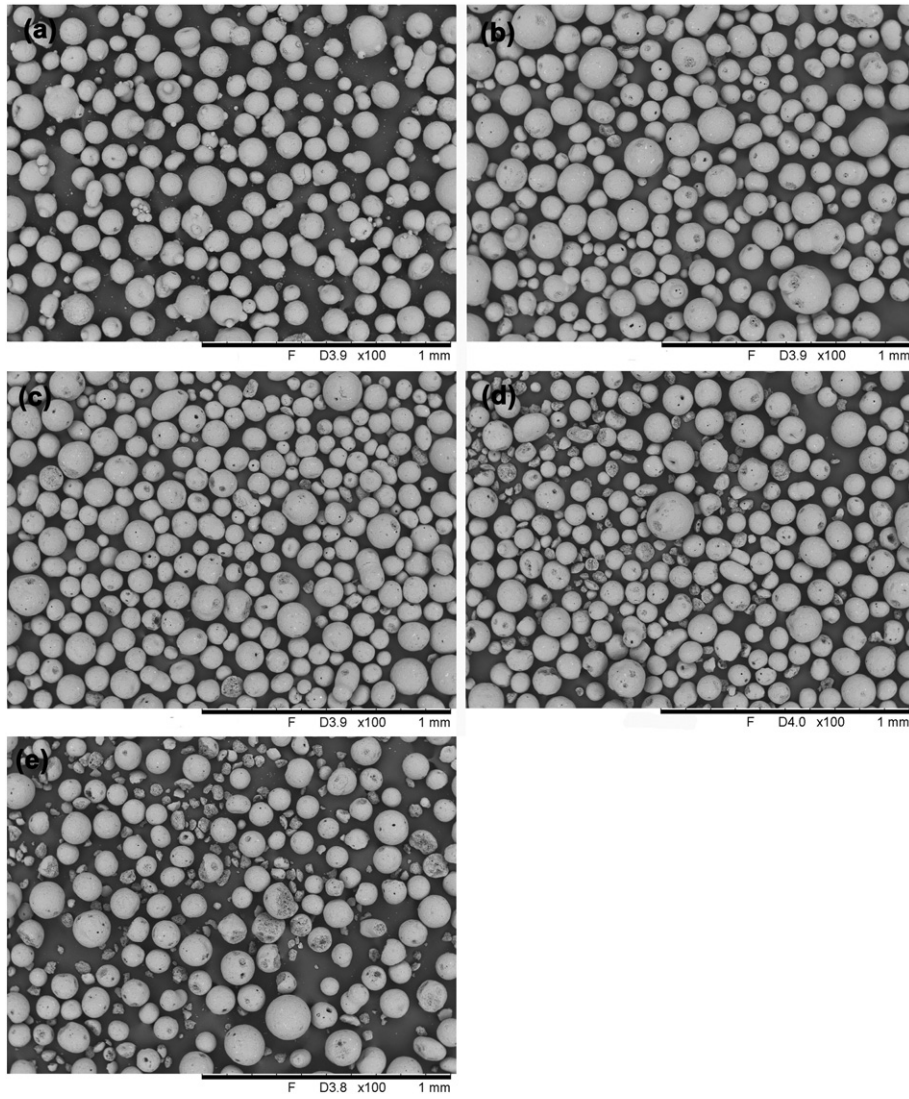


Fig. 6. SEM pictures of the remaining particles in jet cup after tests at room temperature: (a) initial catalyst particles, (b) 88 m/s, (c) 114 m/s, (d) 139 m/s, (e) 158 m/s.

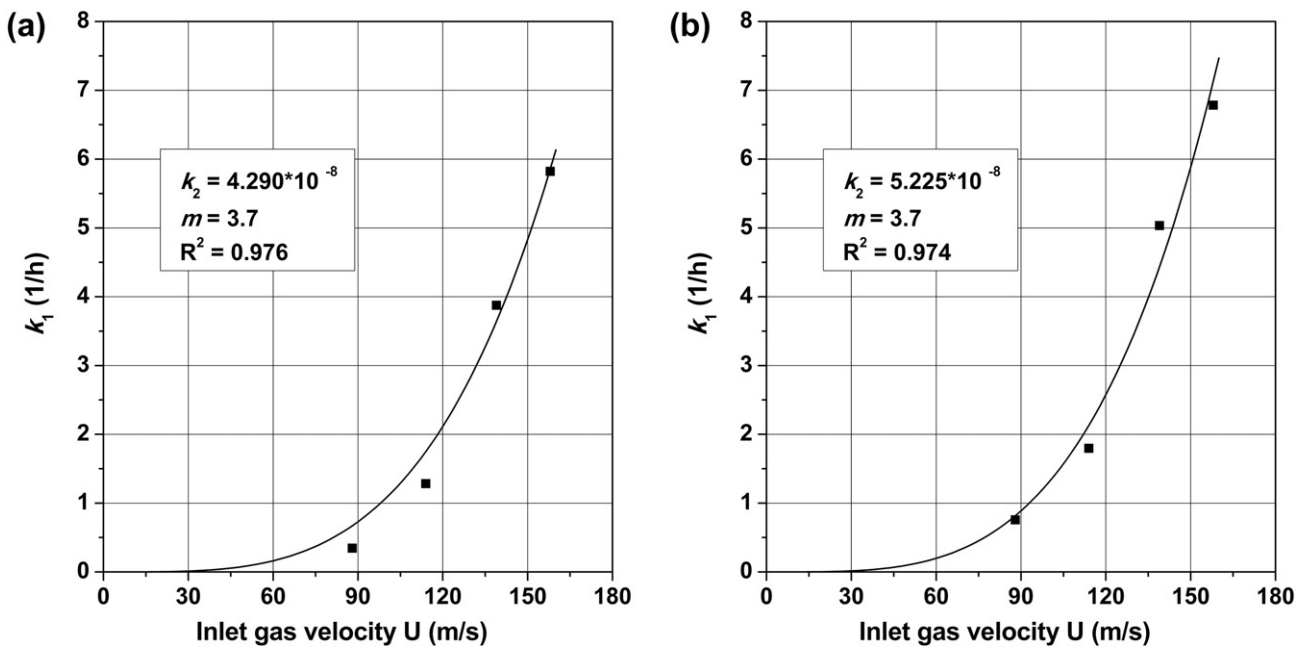


Fig. 7. The influence of inlet gas velocities on attrition rate constant at 100 °C: (a) Al_{20} , (b) Al_{44} .

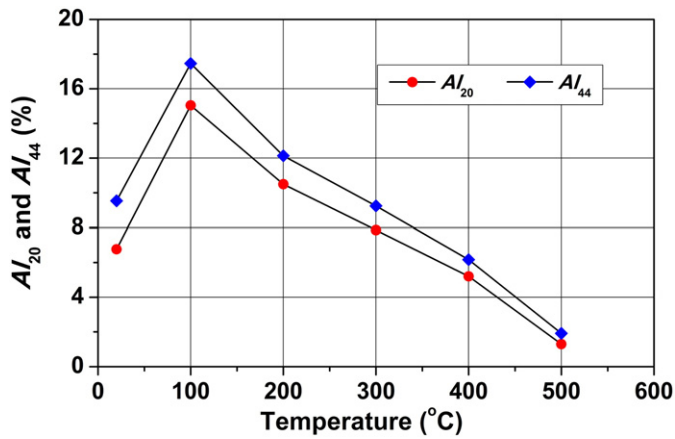


Fig. 8. The influence of temperature on AI. Inlet gas velocity is fixed at 139 m/s, and test time is 3 h.

the inlet gas velocity is increased (158 m/s, as shown in Fig. 6(e)), some small fragments can be visualized in the SEM pictures. This confirmed that the attrition mode at relatively high gas velocity is abrasion with little fragmentation.

Fig. 3 shows the relation between inlet gas velocity and AI. With an increase of inlet gas velocity, the particle velocity will increase and thus the relative velocity between particle and the wall of jet cup also increases. This prompts the friction between particle and the inner wall of jet cup, resulting in a more severe abrasion of particle. We found that the following formulation can best fit the relation between inlet gas velocity and attrition rate constant

$$k_1 = k_2 U^m \quad (2)$$

where k_2 is an attrition rate constant, U is the inlet gas velocity and m is a fitting parameter. Fig. 7 shows the typical results of the attrition rate constant as a function as inlet gas velocity. We obtained $m = 3.7$ in this study. To the knowledge of authors, there is no correlation in the open literature to link the inlet gas velocity with attrition rate in jet cup experiments. Some researchers studied the breakage of FCC catalyst in fluidized beds and related the attrition rate with gas jet velocity [3,30,31]. Werther and Xi [30] used a power index of 3 in their correlation. Kono [31] found that the power index can be 2 or 3, which is dependent on the jet velocity. Boateng et al. [32] argued that the best fitting value of the exponent approaches 4 if the critical velocity is set to 0. The value of the parameter m in our work is in line with these researches.

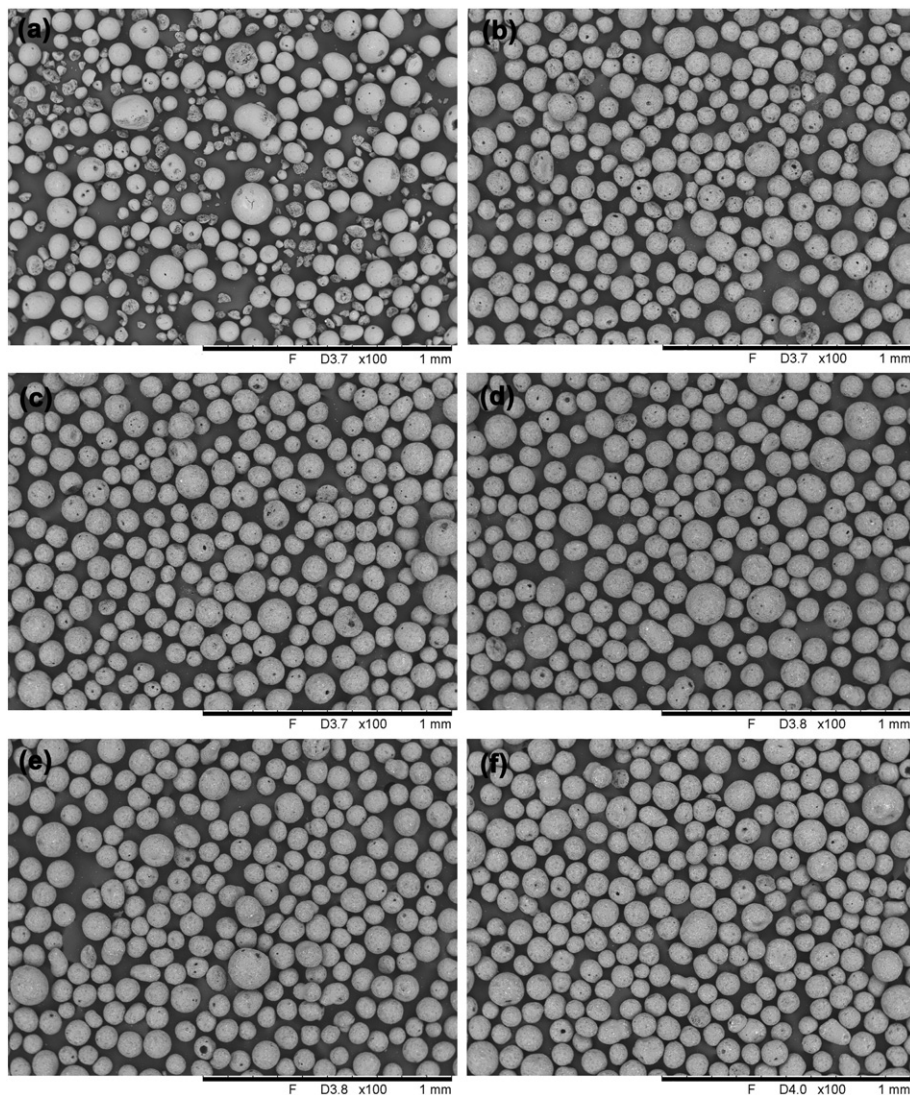


Fig. 9. SEM photos of the remaining particles in jet cup after tests at various temperatures: (a) 20 °C, (b) 100 °C, (c) 200 °C, (d) 300 °C, (e) 400 °C, (f) 500 °C.

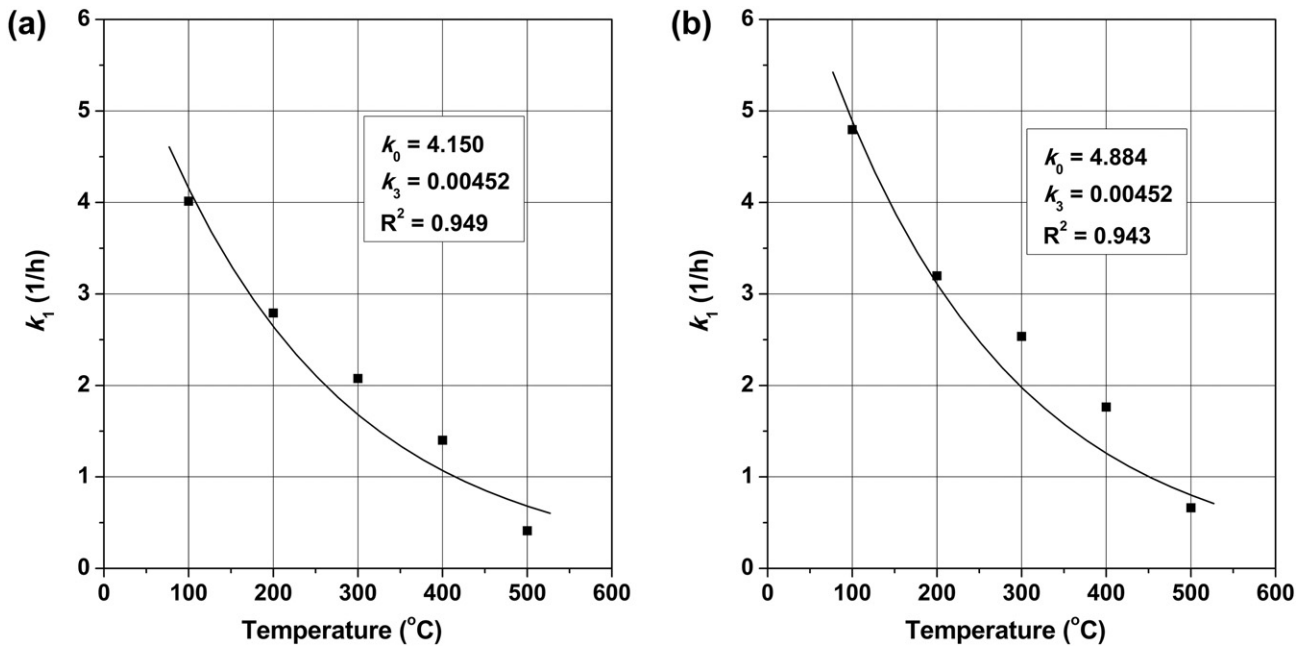


Fig. 10. The influence of temperature on attrition rate constant: (a) Al_{20} , (b) Al_{44} .

3.3. Influence of attrition temperature

Fig. 4 shows the influence of temperature on AI . It can be seen that the AI at any given time interval will decrease as temperature increases from 100 to 500 °C in the jet cup. This may attribute to the change of the properties of gas and catalyst [33,34] at high temperature. However, our results show that the AI (3 hour tests) at 100 °C is larger than that at room temperature, as shown in Fig. 8. This is in accordance with our previous findings in high velocity gas jets experiments [27], in which we demonstrated that the attrition mechanism of MTO catalyst particle at room temperature is different from that at high temperature, and MTO catalyst manifests a maximum AI at round 300 °C in high velocity gas jets [28]. In Fig. 8 we do find such a maximum but it appears at around 100 °C. The reason why the temperature corresponding to the maximum AI shifts from 300 °C in high velocity jets method to 100 °C in jet cup method might be related to the different attrition mechanism. In high velocity gas jets experiments both fragmentation and abrasion of MTO catalyst were found at temperature below 300 °C, and at 300 °C and beyond the abrasion becomes dominant. Fig. 9 shows the SEM pictures for remaining catalyst in the jet cup after tests. At room temperature some fragments can be observed in the sample of remaining particles, as shown in Fig. 9(a). Meanwhile, the mother particles remaining in the jet cup become more smooth and round compared to the initial sample shown in Fig. 6(a). This suggests that both fragmentation and abrasion occur at room temperature in the jet cup. At 100 °C and above, as displayed in Fig. 9(b) to (f), no fragments were found in the sample of remaining particles, which means abrasion becomes dominant. The fragments broken down from the mother particles normally have a large size and are hard to be carried out by gas flow and collected by filter bags. The transition of attrition mechanism is thus likely responsible for maximum AI for MTO catalyst.

In our previous study [28], it is found that the correlations of AI with temperature can be described by an exponential decay function. Here we use the same expression to correlate the attrition rate constant with temperature:

$$k_1 = k_0 e^{-k_3(T-T_0)} \quad (3)$$

where k_0 is the pre-exponential factor, k_3 is constant rate, T_0 is the characteristic temperature. In this study the transition of attrition

mode takes place at 100 °C, and thus we set $T_0 = 100$. The fitting results are shown in Fig. 10.

3.4. Correlation for attrition index of MTO catalyst in jet cup

Based on the analysis above, the influence of various operating conditions on attrition of MTO catalyst in a high temperature jet cup is becoming clear. Following the discussions, we will use Eq. (4) to describe the AI of MTO catalyst in jet cup:

$$AI = k_0 e^{-k_3(T-100)} U^m t^n \quad (4)$$

The parameters retrieved by fitting Eq. (4) using all experimental data are listed in Table 3. In Fig. 11, the AI predicted via Eq. (4) is compared with the experimental data, where a good agreement is obtained.

It should be stressed that, however, the correlation derived in this work needs to be used with caution. First, our experiments were carried out only for commercial MTO catalyst under inlet gas velocity of 88 to 158 m/s and temperature of 100 to 500 °C. The suitability of our correlation for other samples is yet to be experimentally verified. Second, the attrition mechanism of MTO catalyst at high temperature is abrasion, and breakage of particles likely plays a minor role in catalyst attrition process. For the fragmentation dominant attrition process, our correlation may not be applicable.

4. Conclusions

In this work the attrition behavior of MTO catalyst at high temperature was investigated in a jet cup. We studied the influence of test time, inlet gas velocity, and temperature on the attrition index (AI) of MTO

Table 3
The parameters of overall correlation Eq. (4).

Parameters	Al_{20}	Al_{44}
k_0	4.65×10^{-8}	5.43×10^{-8}
k_3	0.00452	0.00452
m	3.7	3.7
n	1.213	1.173
R^2	0.986	0.958

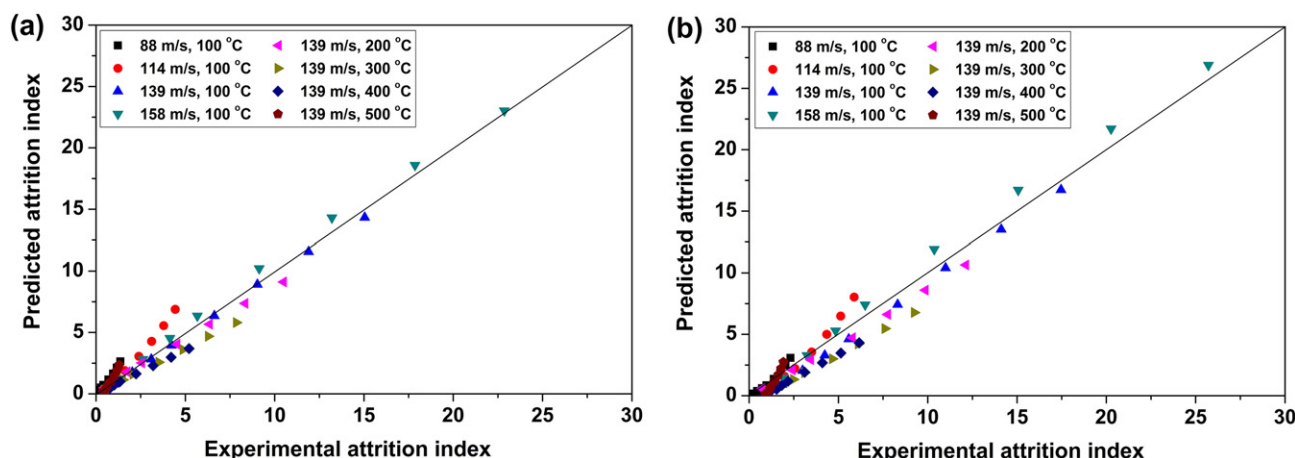


Fig. 11. Comparison between the experimental results and the predicted AI by Eq. (4): (a) Al_2O_3 , (b) Al_4+ .

catalyst. We found that the Gwyn formulation can well represent the relation between AI and test time for MTO catalyst. We compared the parameter n in Gwyn formulation obtained in current jet cup studies with that derived in previous high velocity gas jets experiments. It is found that the deviation is within 2.0% regardless of temperature and attrition test methods. The surprising consistency of n suggests that jet cup method can retrieve quantitatively comparable results with high velocity gas jets method while significantly shortening test time. It is also found that the inlet gas velocity has considerable influence on the MTO catalyst attrition, and the relation between inlet gas velocities and AI can be described by a power index of 3.7. Similar to high velocity gas jets experiments the AI manifests a maximum in jet cup with the increase of temperature. But the temperature corresponding to the maximum AI shifts from 300 °C in high velocity jets tests to 100 °C in jet cup tests. An analysis based on SEM pictures indicates that the transition of attrition mechanism is responsible for this shift. It is shown an exponential decay function can well formulate the relation between AI and temperature. An empirical correlation has been presented for MTO catalyst attrition in jet cup. The correlation can well fit the experimental data for inlet gas velocity from 88 to 158 m/s at temperatures from 100 to 500 °C.

Acknowledgements

This work is supported by the National Natural Science Foundation of China (Grant No. 91334205).

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