碱土金属氧化物对丙三醇和苯胺气相合成 3-甲基吲哚的 Cu/SiO₂-Al₂O₃ 催化剂的作用

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摘要: 研究了碱土金属氧化物的添加对丙三醇和苯胺气相合成 3-甲基吲哚的 Cu/SiO₂-Al₂O₃ 催化剂性能的影响, 采用 X 射线衍射、透射电镜、H₂ 程序升温还原、NH₃ 程序升温脱附以及热重-差热等技术对催化剂进行了表征。结果表明, MgO 能加强活性组分和载体之间的相互作用, 从而促进铜粒子在载体上的分散。另外, MgO 能增加弱酸中心数, 并且抑制积炭的形成, 因而 Cu-MgO/SiO₂-Al₂O₃ 催化剂性能大大提高; 而 CaO, SrO 或 BaO 的加入不利于 Cu/SiO₂-Al₂O₃ 催化剂上 3-甲基吲哚的生成。这是由于铜粒子在载体上的分散度变差, 而且在 Cu-SrO/SiO₂-Al₂O₃ 及 Cu-BaO/SiO₂-Al₂O₃ 催化剂上弱酸中心数较少。碱土金属氧化物的加入不能改变催化剂上积炭的结构。

关键词: 铜; 二氧化硅; 氧化铝; 碱土金属氧化物; 3-甲基吲哚; 丙三醇; 苯胺

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Effect of Alkaline-Earth Metal Oxides on Cu/SiO₂-Al₂O₃ Catalyst for Vapor-Phase Synthesis of 3-Methylindole from Glycerol and Aniline

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Abstract: The effect of the addition of an alkaline-earth metal oxide (MgO, CaO, SrO or BaO) to a Cu/SiO₂-Al₂O₃ catalyst for the vapor-phase synthesis of 3-methylindole from glycerol and aniline was investigated. The catalysts were characterized by X-ray diffraction, transmission electron microscopy, H₂ temperature-programmed reduction, NH₃ temperature-programmed desorption, and thermogravimetric and differential thermal analysis. MgO reinforced the interaction between copper and the support, which promoted the dispersion of Cu particles. In addition, MgO increased the amount of weak acid sites and inhibited the formation of coke. As a result, the addition of MgO to Cu/SiO₂-Al₂O₃ gave a much improved catalyst. Adding CaO, SrO or BaO to Cu/SiO₂-Al₂O₃ deteriorated the catalysts because the dispersion of Cu particles became worse and the amounts of weak acid sites on Cu-SrO/SiO₂-Al₂O₃ and Cu-BaO/SiO₂-Al₂O₃ were fewer. The addition of the alkaline-earth metal oxides did not change the texture of the coke formed.

Key words: copper; silica; aluminum oxide; alkaline-earth metal oxides; 3-methylindole; glycerol; aniline

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3-Methylindole is one of the most important indole derivatives used in industry, agriculture, and medicine [1–5]. It is used in perfumes and in the manufacture of herbicides,

fungicides, dyes, antihypertensive and anticancer medicines [6–8]. In addition, 3-methylindole is an interesting chromophore which is used in biochemistry and analytical

chemistry [9].

Many routes have been developed for the synthesis of 3-methylindole. Among these, the classical Fischer synthesis used for several decades is the most common [10]. Bischler synthesis and Madelung synthesis are also popular routes [11,12]. Although these well-known liquid phase reactions give 3-methylindole in good yield, many drawbacks existed, such as the use of complicated and expensive reagents, a large number of toxic solvents, heavy pollution and severe reaction conditions [13–16]. These limit the application of 3-methylindole. Thus, increasing interest has been focused on the vapor-phase synthesis of 3-methylindole.

The literature is rich in data regarding the vapor-phase synthesis of indoles [17], but scarce for the vapor-phase synthesis of 3-methylindole. Subrahmanyam et al. [18] reported the synthesis of 3-methylindole from indole and methanol (molar ratio of indole/methanol was 1:6) over a Ce/HY zeolite catalyst. The yield of 3-methylindole was 30%. Siwach et al. [12] developed a $\text{Ni}_{1-x}\text{Mn}_x\text{Fe}_2\text{O}_4$ catalyst for the reaction and improved the yield of 3-methylindole up to 54%. Campanati et al. [17] reported a $\text{ZrO}_2/\text{SiO}_2$ catalyst for the synthesis of 3-methylindole from 1,2-propanediol and aniline, but the yield of 3-methylindole was only 12%. Our study found that Ag/SiO_2 was an active catalyst for the reaction and the yield of 3-methylindole reached 35% [19]. However, Ag/SiO_2 showed poor stability. The lifetime of the catalyst dropped rapidly with time.

In recent years, the use of glycerol has become an important research topic in catalysis [20–22]. Glycerol is the main byproduct (about 10 wt%) from biodiesel production, and the effective utilization of glycerol can contribute to the economic production of biodiesel. One of the most attractive approaches is the catalytic conversion of glycerol to high value added chemicals, which agrees with the concept of green chemistry in terms of the utilization of renewable resources.

Very recently, we developed a new approach for the vapor-phase synthesis of 3-methylindole by feeding glycerol and aniline as reactants [23]. Over $\text{Cu}/\text{SiO}_2\text{-Al}_2\text{O}_3$, the yield of 3-methylindole reached 40% and the stability of the catalyst was much better than that of Ag/SiO_2 applied in the conversion of 1,2-propanediol and aniline. Since glycerol, which comes from biomass, is cheap and abundant, and also a green feedstock for the production of various chemicals [21,24], this reaction is a most promising route from the economic and environmental viewpoints.

It is well known that alkaline-earth metal oxides can promote catalytic performance [25–28]. In this paper, an alkaline-earth metal oxide (MgO , CaO , SrO or BaO) was added to $\text{Cu}/\text{SiO}_2\text{-Al}_2\text{O}_3$ for the vapor-phase synthesis of 3-methylindole from glycerol and aniline. X-ray diffraction

(XRD), transmission electron microscopy (TEM), H_2 temperature-programmed reduction (H_2 -TPR), NH_3 temperature-programmed desorption (NH_3 -TPD), and TG-DTA techniques were used to investigate and explain the relationship between the structure of the Cu-based catalysts and their catalytic performance.

1 Experimental

1.1 Catalyst preparation

The $\text{Cu}/\text{SiO}_2\text{-Al}_2\text{O}_3$ catalyst was prepared by incipient wetness impregnation. $\text{SiO}_2\text{-Al}_2\text{O}_3$ (20–40 mesh) was impregnated with an aqueous solution of copper nitrate for 15 h at room temperature, then dried at 120 °C for 4 h and calcined at 500 °C for 4 h to get the catalyst precursor. The $\text{Cu}/\text{SiO}_2\text{-Al}_2\text{O}_3$ catalyst doped with MgO , CaO , SrO or BaO was prepared by sequential impregnation. First, $\text{SiO}_2\text{-Al}_2\text{O}_3$ was pre-impregnated with an aqueous nitrate solution of Mg, Ca, Sr or Ba for 15 h at room temperature, then dried at 120 °C for 4 h and calcined at 500, 600, 800 or 800 °C, respectively, for 4 h to obtain $\text{MO}/\text{SiO}_2\text{-Al}_2\text{O}_3$ ($\text{M} = \text{Mg}$, Ca , Sr or Ba). $\text{MO}/\text{SiO}_2\text{-Al}_2\text{O}_3$ was then impregnated with an aqueous solution of copper nitrate for 15 h, then dried and calcined as with the $\text{Cu}/\text{SiO}_2\text{-Al}_2\text{O}_3$ catalyst described above to obtain the alkaline-earth metal oxide promoted catalyst.

Prior to activity evaluation, 3 ml catalyst was reduced in a gas mixture composed of N_2 (30 ml/min) and H_2 (30 ml/min) at 250 °C for 2 h, then cooled to 240 °C in 0.5 h and kept at this temperature for another 0.5 h. The loading of the active component, copper, was 0.9 mmol/g. The amount of alkaline-earth metal oxide was 0.1 mmol/g.

1.2 Catalytic activity measurement

The catalytic reaction was carried out in a fixed bed continuous flow glass reactor with an inside diameter of 12 mm under atmospheric pressure. The reactor was placed vertically inside a tubular furnace and heated electrically. The reactor temperature was monitored by a thermocouple with its tip located at the catalyst bed and connected to a temperature controller. The solution of reactants (molar ratio of aniline/glycerol = 3:1) was pumped through the preheater where it was vaporized and the reactants were carried into the reactor by flowing H_2 (10 ml/min), steam (12 ml/h), and N_2 (58 ml/min). The reaction was carried out at 240 °C. The quantitative analysis of the reactants and products was carried out on a SP-6890A gas chromatograph equipped with a SE-54 capillary column. 1-Hexyl alcohol was used as an internal standard.

1.3 Catalyst characterization

XRD pattern was recorded using a D8 advance diffraction meter with a Cu K_{α} radiation source at a scan rate of 0.5°/min in the 2θ range of 30°–80°. The voltage and current were 40 kV and 40 mA, respectively. TEM experiments were done on a JEM-2000EX electron microscope at the accelerating voltage of 50 kV. The samples were crushed and dispersed in alcohol and then spread on a holey carbon Cu microgrid. The particle size distribution was obtained from several micrographs. The average particle size was estimated using the equation $d = \sum(n_i d_i) / \sum n_i$, where n_i was the number of particles with diameter d_i and $\sum n_i$ was the number of particles used to compute the size distribution.

H₂-TPR experiments were carried out in a quartz reactor with inside diameter of 6 mm and length of 350 mm. A 100 mg sample was pretreated in a flow of ultrapure nitrogen gas of 50 ml/min at 100 °C for 1 h to remove water and other contaminants. TPR profiles were obtained by heating the sample under a 10% H₂/Ar flow (50 ml/min) from 50 to 450 °C at a linearly programmed rate of 10 °C/min. The hydrogen consumption was recorded with a thermal conductivity detector. NH₃-TPD was carried out in the quartz reactor used in H₂-TPR experiments. A sample of 150 mg was pretreated at 500 °C for 1 h in a flow of ultrapure helium gas (30 ml/min) to remove water and other contaminants from the catalyst and cooled down to 100 °C, then saturated with ammonia gas at 100 °C. After the sample was purged with helium gas (30 ml/min) at 100 °C for 2 h to remove physisorbed ammonia, TPD was carried out from 100 to 700 °C with a temperature ramp of 10 °C/min.

TG-DTA analysis was carried out with a TG/DTA 6300 apparatus to characterize the coke on the catalyst. The used catalyst sample (7.8 mg) was placed in the pan. The sample was first heated in ultrapure nitrogen (20 ml/min) from room temperature to 300 °C at a heating rate of 10 °C/min to remove physisorbed species. After keeping at 50 °C, it was heated in air (20 ml/min) to 800 °C at a heating rate of 10 °C/min.

2 Results and discussion

2.1 Catalyst characterization

2.1.1 XRD and TEM results

The XRD patterns of the fresh and used catalyst are shown in Fig. 1. Two diffraction peaks of copper at 2θ of 43.3° and 50.5°, assigned to the (111) and (200) reflections [29], were observed on fresh Cu/SiO₂-Al₂O₃. The same diffraction peaks were seen on fresh Cu-MgO/SiO₂-Al₂O₃, but the intensity was a little weaker. On adding CaO or SrO to Cu/SiO₂-Al₂O₃, the intensity of the diffraction peaks became stronger. With fresh Cu-BaO/SiO₂-Al₂O₃, another

Fig. 1. XRD patterns of the fresh and used catalysts. (1) Fresh Cu/SiO₂-Al₂O₃; (2) Fresh Cu-MgO/SiO₂-Al₂O₃; (3) Fresh Cu-CaO/SiO₂-Al₂O₃; (4) Fresh Cu-SrO/SiO₂-Al₂O₃; (5) Fresh Cu-BaO/SiO₂-Al₂O₃; (6) Used catalysts of Cu/SiO₂-Al₂O₃; (7) Used Cu-MgO/SiO₂-Al₂O₃; (8) Used Cu-CaO/SiO₂-Al₂O₃; (9) Used Cu-SrO/SiO₂-Al₂O₃; (10) Used Cu-BaO/SiO₂-Al₂O₃.

diffraction peak at 74.7°, assigned to the (220) reflection of copper, was observed and the intensity of the diffraction peaks was much stronger than that from fresh Cu/SiO₂-Al₂O₃.

On the used Cu-based catalysts, three diffraction peaks at 2θ of 43.3°, 50.5°, and 74.7° were observed, and the intensity of the copper diffraction peaks from Cu/SiO₂-Al₂O₃ and the samples doped with MgO, CaO, or SrO became stronger.

Figure 2 is TEM images of the fresh and used Cu-based catalysts. The average copper particle sizes obtained from these are shown in Table 1. The copper particle size of fresh Cu-MgO/SiO₂-Al₂O₃ was smaller than that of fresh Cu/SiO₂-Al₂O₃. On fresh Cu/SiO₂-Al₂O₃ doped with CaO, SrO or BaO, however, the copper particle sizes were much larger than that of fresh Cu/SiO₂-Al₂O₃. On the used

Table 1 Average copper particle sizes of the Cu-based catalysts

Catalyst	Average copper particle size (nm)	
	Fresh	Used
Cu/SiO ₂ -Al ₂ O ₃	15	20
Cu-MgO/SiO ₂ -Al ₂ O ₃	13	18
Cu-CaO/SiO ₂ -Al ₂ O ₃	23	34
Cu-SrO/SiO ₂ -Al ₂ O ₃	24	36
Cu-BaO/SiO ₂ -Al ₂ O ₃	35	38

Fig. 2. TEM images of fresh and used catalysts. (a) Fresh Cu/SiO₂-Al₂O₃; (b) Fresh Cu-MgO/SiO₂-Al₂O₃; (c) Fresh Cu-CaO/SiO₂-Al₂O₃; (d) Fresh Cu-SrO/SiO₂-Al₂O₃; (e) Fresh Cu-BaO/SiO₂-Al₂O₃; (f) Used catalysts of Cu/SiO₂-Al₂O₃; (g) Used Cu-MgO/SiO₂-Al₂O₃; (h) Used Cu-CaO/SiO₂-Al₂O₃; (i) Used Cu-SrO/SiO₂-Al₂O₃; (j) Used Cu-BaO/SiO₂-Al₂O₃.

Cu-based catalysts, the copper particle sizes became larger compared with those of the fresh catalysts.

From the results in Figs. 1 and 2 and Table 1, it can easily be seen that MgO promoted the dispersion of copper particles on the support. The addition of CaO, SrO, or BaO to Cu/SiO₂-Al₂O₃ brought about a poorer dispersion of Cu particles. There was sintering of copper particles during the reaction. Adding alkaline-earth metal oxides to Cu/SiO₂-Al₂O₃ did not inhibit the sintering of Cu particles.

2.1.2 H₂-TPR results

The reduction behavior of CuO/SiO₂-Al₂O₃ and the alkaline-earth metal oxide doped sample was investigated by H₂-TPR. The profiles are shown in Fig. 3. No hydrogen consumption peak was seen in the temperature range of 150–400 °C with the MO/SiO₂-Al₂O₃ (M = Mg, Ca, Sr or Ba) samples, which indicated that the alkaline-earth metal oxides were not reduced in this temperature range. The reduction peak of CuO was shifted towards higher temperature after adding MgO to CuO/SiO₂-Al₂O₃, which implied that there existed a strong interaction between CuO and the support that resulted in a better dispersion of CuO particles on the support and made the reduction of CuO more difficult. The reduction peaks of CuO were shifted towards a lower temperature in the order of CaO, SrO and BaO, which revealed that the interaction between CuO and the support became weaker, and as a result, CuO particles agglomerated and were reduced easily [30].

Fig. 3. H₂-TPR profiles of MgO/SiO₂-Al₂O₃ (1), CaO/SiO₂-Al₂O₃ (2), SrO/SiO₂-Al₂O₃ (3), BaO/SiO₂-Al₂O₃ (4), CuO/SiO₂-Al₂O₃ (5), CuO-MgO/SiO₂-Al₂O₃ (6), CuO-CaO/SiO₂-Al₂O₃ (7), CuO-SrO/SiO₂-Al₂O₃ (8), and CuO-BaO/SiO₂-Al₂O₃ (9).

2.1.3 NH₃-TPD results

The desorption of NH₃ provides information about the distribution and amount of acid sites on the catalyst. The NH₃-TPD profiles of Cu/SiO₂-Al₂O₃ and the alkaline-earth metal oxide doped catalysts are shown in Fig. 4. Three desorption peaks were observed. The peak at the lower temperature (< 220 °C) was assigned to weak acid sites, while the others at the higher temperatures (220–450 °C) corre-

Fig. 4. NH₃-TPD profiles of Cu/SiO₂-Al₂O₃ (1), Cu-MgO/SiO₂-Al₂O₃ (2), Cu-CaO/SiO₂-Al₂O₃ (3), Cu-SrO/SiO₂-Al₂O₃ (4), and Cu-BaO/SiO₂-Al₂O₃ (5).

sponded to the medium-strong acid sites. After adding MgO to Cu/SiO₂-Al₂O₃, the amount of weak acid sites increased, and the amount of the medium-strong acid sites decreased. On Cu-CaO/SiO₂-Al₂O₃, both the amounts of weak acid sites and medium-strong acid sites increased. On Cu-SrO/SiO₂-Al₂O₃ and Cu-BaO/SiO₂-Al₂O₃, the amount of the weak acid sites decreased, but the medium-strong acid sites were increased.

2.1.4 TG-DTA analysis

Many investigations showed that coking during the reaction is one of the reasons for the deactivation of the catalyst [31]. TG-DTA analysis was used to investigate the formation of the coke. TG-DTA results of used Cu/SiO₂-Al₂O₃ and alkaline-earth metal oxide doped catalysts are shown in Fig. 5. An obvious mass loss was observed, suggesting the formation of coke during the reaction. Since the mass loss from used Cu/SiO₂-Al₂O₃ doped with MgO, SrO or BaO decreased compared with that from used CuO/SiO₂-Al₂O₃, it can be deduced that the alkaline-earth metal oxides inhibited the formation of coke. The mass loss increased a little on Cu-CaO/SiO₂-Al₂O₃, which indicated that CaO did not suppress the formation of coke. From Figs. 4 and 5(a), it is easily found that the amount of the coke was correlated with the amount of acid sites on the catalysts. With more acid sites, there was more coke.

In Fig. 5(b), two exothermic peaks were observed, which revealed that there existed two types of coke. The exothermic peak at 355 °C was assigned to the removal of easily oxidized carbon. Another exothermic peak at 470 °C was the oxidation of regular or graphitic carbon. There was no obvious shift for the exothermic peaks on used

Fig. 5. TG (a) and DTA (b) profiles of used catalysts of Cu/SiO₂-Al₂O₃ (1), Cu-MgO/SiO₂-Al₂O₃ (2), Cu-CaO/SiO₂-Al₂O₃ (3), Cu-SrO/SiO₂-Al₂O₃ (4), and Cu-BaO/SiO₂-Al₂O₃ (5).

Cu/SiO₂-Al₂O₃ doped with the alkaline-earth metal oxides, which indicated that the addition of the alkaline-earth metal oxides to Cu/SiO₂-Al₂O₃ did not change the texture of the coke.

2.2 Activity of Cu/SiO₂-Al₂O₃ doped with alkaline-earth metal oxides

The effect of alkaline-earth metal oxide additives on the performance of the Cu/SiO₂-Al₂O₃ catalyst for the vapor-phase synthesis of 3-methylindole from glycerol and aniline is shown in Fig. 6. Over Cu/SiO₂-Al₂O₃, glycerol conversion decreased with time on stream and 3-methylindole yield increased during the first 3 h, and then dropped. A similar phenomenon was seen over Cu/SiO₂-Al₂O₃ doped with MgO, CaO, SrO or BaO, although the activity and stability were rather different. Over Cu-MgO/SiO₂-Al₂O₃, glycerol conversion and 3-methylindole yield were higher than that over Cu/SiO₂-Al₂O₃ and the stability was improved. The yield of 3-methylindole was up to 46% at the third hour of reaction. Over Cu-CaO/SiO₂-Al₂O₃, although the initial activity was higher than that over Cu/SiO₂-Al₂O₃, the stability was poor, and the final yield of 3-methylindole was lower than that over Cu/SiO₂-Al₂O₃. After adding SrO or BaO to Cu/SiO₂-Al₂O₃, both activity and stability decreased.

For the vapor-phase synthesis of 3-methylindole from

Fig. 6. Glycerol conversion (a) and 3-Methylindole yield (b) versus time on stream over Cu/SiO₂-Al₂O₃ (1), Cu-MgO/SiO₂-Al₂O₃ (2), Cu-CaO/SiO₂-Al₂O₃ (3), Cu-SrO/SiO₂-Al₂O₃ (4), and Cu-BaO/SiO₂-Al₂O₃ (5).

aniline and glycerol, it has been found that a Cu-based catalyst with good dispersion and large amount of weak acid sites was superior for the production of 3-methylindole [25]. The characterization results showed that MgO enhanced the interaction between the active component of copper and the support, which promoted the dispersion of Cu particles on the support. In addition, MgO increased the amount of weak acid sites and decreased the amount of the medium-strong acid sites, which helped the adsorption of reactants on the catalyst and the desorption of 3-methylindole from the catalyst. Moreover, MgO helped suppressing the formation of coke. As a result, the addition of MgO promoter improved the performance of the catalyst remarkably. Adding CaO to Cu/SiO₂-Al₂O₃ increased the amount of the weak acid sites, which enhanced the initial activity of the catalyst, but it brought about a poor dispersion of Cu particles on the support and a larger amount of the medium-strong acid sites, which resulted in a low final activity and poor stability of the catalyst. On Cu-SrO/SiO₂-Al₂O₃ or Cu-BaO/SiO₂-Al₂O₃, the dispersion of Cu particles became worse and the amount of the weak acid sites was much less than that on Cu/SiO₂-Al₂O₃, and as a result, the activity of the catalyst was low and the stability was poor.

3 Conclusions

The addition of MgO to Cu/SiO₂-Al₂O₃ enhanced the ac-

tivity and stability of the catalyst for the vapor-phase synthesis of 3-methylindole from glycerol and aniline. The yield of 3-methylindole was increased to 46% after three hour of reaction. The promoting effect of MgO stemmed from its ability to improve the dispersion of copper particles and increase the amount of weak acid sites, as well as suppressing the formation of coke during the reaction. The addition of CaO, SrO or BaO to Cu/SiO₂-Al₂O₃ decreased the dispersion of copper particles, which resulted in poorer activity and stability, although SrO or BaO can inhibit the formation of coke. The addition of the alkaline-earth metal oxides to Cu/SiO₂-Al₂O₃ did not change the texture of the coke.

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根据美国汤森路透 (Thomson Reuters) 2012 年 6 月 29 日公布的 2011 年度《期刊引证报告》(*Journal Citation Reports*, JCR), 《催化学报》2011 年 SCI 影响因子首次突破 1.0, 达到 1.171, 总被引频次为 1529, 5 年影响因子为 0.945. 其中, 影响因子和 5 年影响因子均位居 SCI 收录的中文化学类期刊第 1 名, 也是历年来我国中文化学类期刊 SCI 影响因子的最高值. 这表明, 《催化学报》正在被越来越多的国内外同行阅读和参考, 该刊在国际学术交流中的作用进一步提高. 《催化学报》取得的成绩离不开广大作者、审稿专家和读者多年来的大力支持和帮助, 在此向您表示衷心感谢!

(《催化学报》编辑部 2012-07-02)

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(Editorial Office of *Chinese Journal of Catalysis*, 2012-07-02)