

## 常压下 Pt-Bi 双金属催化剂上甘油选择性氧化

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**摘要:** 制备了一系列活性碳 (AC) 负载的 Pt-Bi 双金属催化剂, 考察了催化剂中 Bi 含量对其催化甘油选择性氧化反应性能的影响。结果表明, 适量 Bi 的添加可以改善催化剂中 Pt 的氧化还原性能, 从而有利于催化剂活性的提高和二羟基丙酮 (DIHA) 产物的生成。当 Bi 的含量为 5% 时, 该催化剂的活性最高, 甘油转化率和 DIHA 选择性分别达到 91.5% 和 49.0%。表征结果显示, Pt-Bi 颗粒的平均粒径为 3.8 nm, 且高度分散在催化剂表面, 这是该催化剂具有较高活性的主要原因。

**关键词:** 甘油氧化; 铂; 铋; 活性碳载体; 二羟基丙酮

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## Glycerol Oxidation with Oxygen over Bimetallic Pt-Bi Catalysts under Atmospheric Pressure

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**Abstract:** A series of bimetallic Pt-Bi catalysts with a constant platinum content of 5.0 wt% and a varied bismuth content (3.0–7.0 wt%) supported on active carbon were prepared and used for glycerol oxidation with oxygen under atmospheric pressure. The bimetallic Pt-Bi/C was efficient for the selective oxidation of glycerol to dihydroxyacetone (DIHA) and the selectivity for DIHA reached 49.0% at a 91.5% conversion of glycerol over the 5%Pt-5%Bi/C catalyst. X-ray diffraction and transmission electron microscopy analysis revealed that the specially configured Pt-Bi nanoparticles in 5%Pt-5%Bi/C were highly dispersed (3.8 nm) over the active carbon support, which is proposed to contribute to the improved performance.

**Key words:** glycerol oxidation; platinum; bismuth; carbon support; dihydroxyacetone

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The demand for alternative renewable fuels has grown because of the progressive depletion of fossil resources and the increased use of the world's energy resources [1]. Biodiesel, a well-known alternative fuel for diesel engines [2], can be produced from renewable biological sources such as

vegetable oils by transesterification during which glycerol is an inevitable byproduct [3–5]. The resulting excess surplus glycerol produced because of the increased expansion of biodiesel production drives researchers to investigate new applications for this highly versatile compound, which is a

potential starting material for numerous value-added products [6,7]. As glycerol is a highly functionalized molecule compared with hydrocarbons, one advantageous alternative is to use it as a feedstock for the production of valuable oxygenated derivatives [7–9].

Among various important compounds that can be produced by the selective oxidation of glycerol, dihydroxyacetone (DIHA) is one of the most interesting because of its applications in cosmetics and in the fine chemicals industry. In particular, it is used as an active ingredient in sunless tanning agents [7,8]. DIHA is currently produced from the microbial fermentation of glycerol by *Gluconobacter oxydans*. However, productivity is low because of the low substrate (glycerol) concentration and the long fermentation time [10]. Recently, the electrocatalytic oxidation of glycerol with tetramethyl piperidine-1-oxyl (TEMPO) as the oxidant and Ag/AgCl as the catalyst was investigated by Ciriminna et al. [8,11]. They found that the yield of DIHA reached 25% at 20 h, however, the over-oxidation of DIHA to hydroxypyruvic was observed at 200 h [11]. The selective oxidation of glycerol to DIHA by a heterogeneous process with a supported metal as the catalyst, water as the solvent, and air as the oxidizing agent is highly appealing because of its economic and clean chemistry features (non-polluting, non-toxic effluents, recyclable catalyst) [7,9,12].

Recently Kimura et al. [12] found that platinum supported on charcoal (Pt/C) showed weak catalytic activity for the oxidation of the secondary hydroxy group of glycerol especially in an acidic medium (pH 2–4) where the DIHA yield is only 4%. Because of the poor performance of these Pt/C catalysts they focused on the modification of the Pt/C catalysts and the optimization of reaction conditions. The best catalytic performance was observed when bismuth was used as a promoter, and the selectivity for DIHA increased from 10% to 80% when a low conversion of glycerol (40%) was used over a bismuth modified platinum catalyst in a fixed-bed reactor [13]. In batch reactors the highest yield obtained for DIHA was 37% at a 75% glycerol conversion, as reported by Garcia et al. [9] over a carbon supported Pt-Bi catalyst that was prepared by co-impregnation. A Au/C catalyst was also active in the liquid-phase oxidation of glycerol to DIHA (26% selectivity) under atmospheric pressure if a base was added to the reaction mixture (NaOH/glycerol > 2). However, the product distribution of glycerol oxidation over the Au/C catalyst depended strongly on the base concentration and the reaction time and the main product was glyceric acid (44%) [7,14]. The selectivity for DIHA increased from 26% to 36% at 50% glycerol conversion by alloying gold with platinum [14].

It has also been suggested that bismuth can act as a site blocker on Pt ensembles [12,15], which suppresses the irreversible adsorption of the formed by-products (e.g. glyceric

acid), which would otherwise lead to catalyst deactivation during the oxidation of secondary alcohols [16–18]. On the other hand, Besson et al. [19,20] indicated that bismuth atoms can adsorb oxygen to form Pt–Bi–OH species on the surface, which acts as a new active site for the oxidation of secondary alcohols adsorbed onto platinum. Hu et al. [21] reported a systematic investigation into the selective oxidation of glycerol to DIHA in a semi-batch reactor over a Pt-Bi/C catalyst with different metal loadings and preparation methods under high oxygen pressures.

In this work, bimetallic Pt-Bi catalysts with a constant platinum content of 5.0 wt% and different bismuth contents (3.0, 5.0, and 7.0 wt%) supported on active carbon (AC) were prepared and used for the selective oxidation of glycerol by molecular oxygen at atmospheric pressure. The morphology and structure of these catalysts were characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM).

## 1 Experimental

### 1.1 Catalyst preparation

First, AC (9.3  $\mu\text{m}$  average size, Tangshan Jianxin Active Carbon Co., Ltd., China) was pretreated with hydrogen peroxide at 50 °C for 5 h and washed with distilled water. The suspension was then filtered and dried overnight at 60 °C in vacuum. Second, 2.5 ml of platinum chloride ( $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ , > 99%, Shanghai Chemical Reagent Co., Ltd., China) solution (0.1 g/ml) was added dropwise to a stirred suspension of 5.0 g AC at 50 °C followed by the addition of 4.0 ml formaldehyde (37%, Shanghai Chemical Reagent Co., Ltd., China). The mixture was then stirred at 50 °C for 1 h at a final pH of 9.0. The slurry was filtered and washed with distilled water until it was free of chloride. This catalyst, denoted Pt/C with a loading amount of 5.0 wt% Pt as confirmed by inductively coupled plasma-atomic emission spectroscopy (ICP, plasma-Spec-II spectrometer), was kept in moisture before use.

The prepared Pt/C was further impregnated with a predetermined amount of  $\text{BiCl}_3$  (> 99.99%, Shanghai Chemical Reagent Co., Ltd., China) solution and dried in vacuum followed by hydrogen reduction at 250 °C and heat treatment at 500 °C under a nitrogen flow (40 ml/min). Catalysts with a 3.0, 5.0, and 7.0 wt% Bi loading were prepared following the same protocol and they are denoted 5%Pt-3%Bi/C, 5%Pt-5%Bi/C, and 5%Pt-7%Bi/C, respectively. For comparison, a Bi/C catalyst with a 5.0 wt% loading of Bi supported on AC was synthesized using the same impregnation method as described above.

### 1.2 Catalyst characterization

The catalysts were characterized by XRD and TEM. XRD analysis was performed on a Rigaku automated power X-ray diffractometer system (RINT 2500, Japan) with Cu  $K_{\alpha}$  radiation at 45 kV and 100 mA. Diffraction data were recorded using continuous scanning at  $0.02^{\circ}/s$ , step  $0.02^{\circ}$ . TEM images and an energy dispersive X-ray (EDX) spectra were obtained using an accelerating voltage of 200 kV (TEM, JEOL-2020F). Powder samples of the catalysts were ultrasonicated in ethanol and dispersed on Cu grids.

### 1.3 Glycerol oxidation

Glycerol (> 99.0%) solution (50 ml, 0.1 g/ml) and 0.5 g of the catalyst were added to a reactor. Oxygen (150 ml/min) was introduced to the suspension when the desired temperature was reached. After the reaction, the catalyst was removed by filtration and the aqueous solution was analyzed by a high-performance liquid chromatograph equipped with a refractive index (RI) detector and a Zorbax SAX column (HPLC, Agilent 1100 series). All products in the reaction mixture were quantified using an external calibration method. During the oxidation reaction, the gas in the effluent was collected and analyzed using the TCD gas chromatograph equipped with an APS-201 (Flusin T) column. Detailed analytical procedures for these products have been described previously [22–25].

## 2 Results and discussion

### 2.1 Characterization results

Figure 1 shows XRD patterns of the carbon supported Pt and Pt-Bi nanoparticles. The peaks in the spectrum of Pt/C (curve (1)) at  $2\theta = 40.3^{\circ}$ ,  $46.5^{\circ}$ ,  $67.7^{\circ}$ , and  $81.6^{\circ}$  are assigned to the (111), (200), (220), and (311) phases of the platinum crystal, respectively. The broad peak centered at  $2\theta = 24.6^{\circ}$  comes from the diffraction of the active carbon support (JCPDS 75-0223). The diffraction peaks of Pt in 5%Pt-3%Bi/C (curve (2)) become faint and weak, which indicates that Bi improved the dispersion of Pt. However, a different XRD pattern was observed for 5%Pt-5%Bi/C (curve (3)) and 5%Pt-7%Bi/C (curve (4)). Bismuth carbonate oxide ( $\text{Bi}_2\text{O}_2\text{CO}_3$ ) was detected in 5%Pt-5%Bi/C (JCPDS 41-1488) and its reflexes were further enhanced in 5%Pt-7%Bi/C (curve (4)). On the other hand, the intensity of the platinum reflexes became weaker with the increase of Bi content. The mean particle sizes of the platinum calculated by the Scherrer-Warren equation were 3.4, 2.9, and 1.0 nm for Pt/C, 5%Pt-3%Bi/C, and 5%Pt-5%Bi/C, respectively. These results indicate that the dispersion of Pt was improved by Bi and well-dispersed Pt of fine grain size can be achieved when the amount of Bi loading is higher than

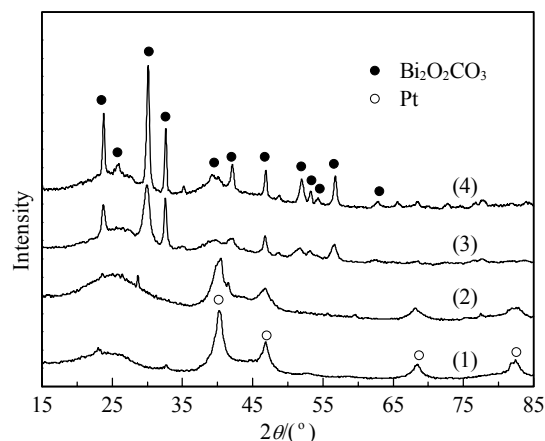


Fig. 1. XRD patterns of Pt/C (1), 5%Pt-3%Bi/C (2), 5%Pt-5%Bi/C (3), and 5%Pt-7%Bi/C (4).

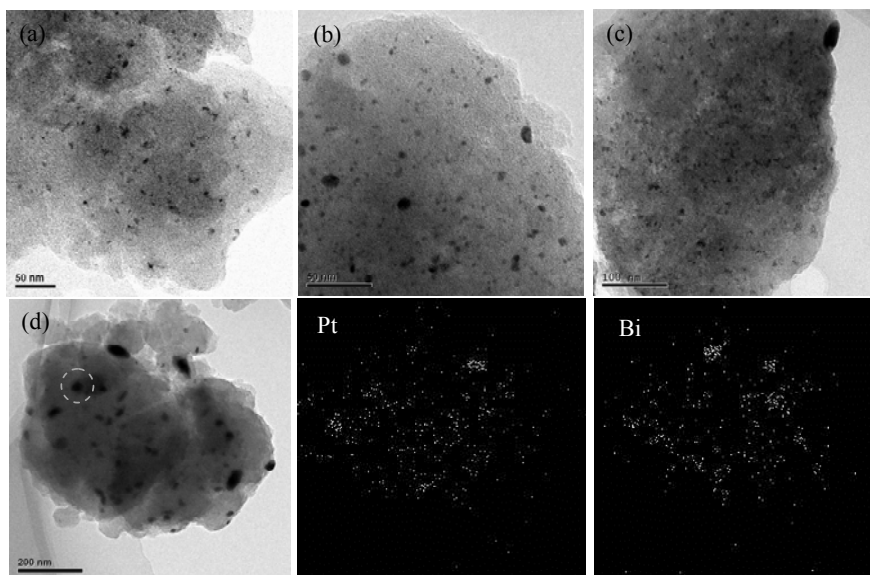
5%.

Representative micrographs of the Pt/C, 5%Pt-3%Bi/C, 5%Pt-5%Bi/C, and 5%Pt-7%Bi/C catalysts along with the corresponding particle distribution and EDX spectra are shown in Fig. 2. The average particle size was calculated by

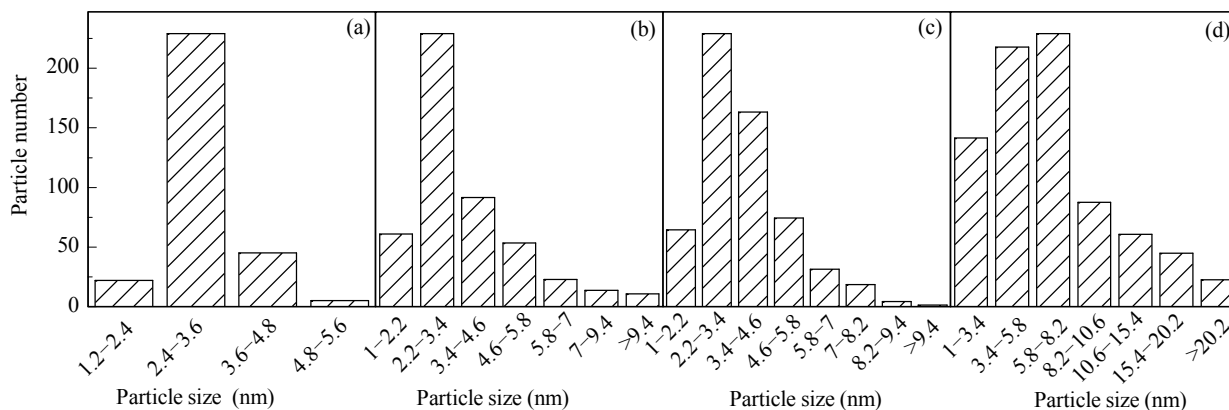
$$d = \frac{\sum n_i d_i^3}{\sum n_i d_i^2}$$

where  $n_i$  is the number of particles having a diameter  $d_i$ . In addition, at least 300 particles from pictures of each catalyst were measured and the data are shown in Fig. 3. Highly dispersed Pt particles with a mean particle size of 3.1 nm were detected in Pt/C (Fig. 2(a) and Fig. 3(a)). Comparatively, larger sized metal particles with higher dispersion were detected in bimetallic Pt-Bi/C and the corresponding EDX spectra revealed the presence of both Pt and Bi in the nano-composition of each sample (Fig. 4). The particle size distribution was fairly narrow in 5%Pt-3%Bi/C, and the majority of particles ranged from 2.2–4.6 nm with a mean particle size of 3.7 nm (Fig. 2(b) and Fig. 3(b)). 5%Pt-5%Bi/C (Fig. 2(c) and Fig. 3(c)) had a similar mean particle size (3.8 nm) whereas the distribution of the detected nanoparticles became broader (1.0–5.8 nm) than that of 5%Pt-3%Bi/C. 5%Pt-7%Bi/C comprised particles of 1.0–10.6 nm in size with a significant number of larger particles over 10.6 nm (Fig. 2(d) and Fig. 3(d)), which resulted in an increase of its mean particle size to 7.1 nm. EDX elemental mapping (Fig. 2(d)) provided insight into the nano-composition and the elemental distribution in the random large particles in 5%Pt-7%Bi/C. Visual evidence was obtained to show that Bi and Pt are homogeneously distributed within the large particles.

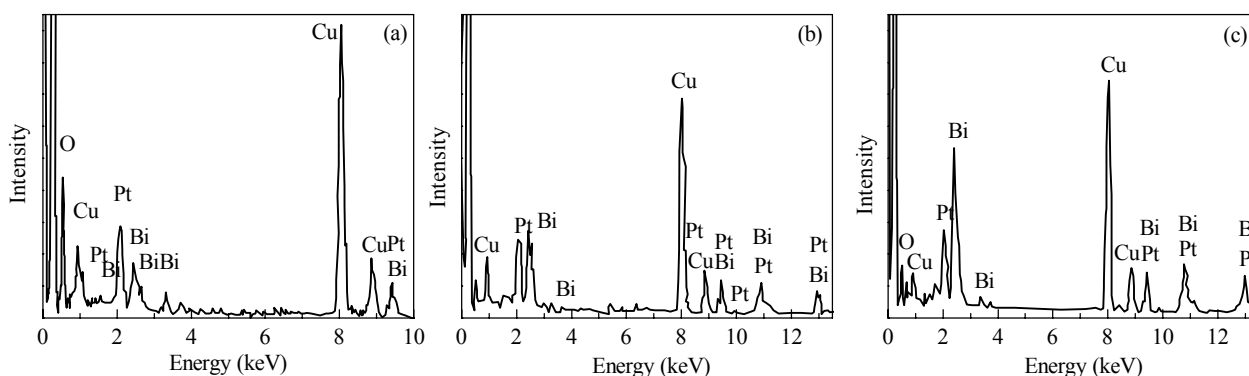
The above-mentioned XRD analysis indicated that the dispersion of Pt was improved by the added Bi (Table 1) but the outline of the Pt-Bi particles in the TEM images of 5%Pt-5%Bi/C and 5%Pt-7%Bi/C increased. These contra-



**Fig. 2.** TEM images of Pt/C (a), 5%Pt-3%Bi/C (b), 5%Pt-5%Bi/C (c), and 5%Pt-7%Bi/C (d) and the elemental mapping images for Pt and Bi in a random nanoparticle of 5%Pt-7%Bi/C



**Fig. 3.** Particle size distribution of Pt/C (a), 5%Pt-3%Bi/C (b), 5%Pt-5%Bi/C (c), and 5%Pt-7%Bi/C (d) according to their TEM images.



**Fig. 4.** EDX spectra of 5%Pt-3%Bi/C (a), 5%Pt-5%Bi/C (b), and 5%Pt-7%Bi/C (c).

dictory trends are attributed to the fact that the Pt particles were wrapped by Bi in the Pt-Bi particles. A schematic diagram of these wrapped Pt-Bi particles is shown in Fig. 5. Kimura et al. [12] once suggested that Bi atoms function as site blockers on Pt(111) and a Bi sub-monolayer on Pt (111) with a ( $\sqrt{3}\times\sqrt{3}$ ) structure would facilitate the formation of

DIHA in glycerol.

## 2.2 Catalytic activity

A monometallic Bi/C catalyst was found to be inactive toward the oxidation of glycerol at atmospheric pressure

**Table 1** Oxidation of glycerol using carbon supported monometallic Pt and bimetallic Pt-Bi catalysts

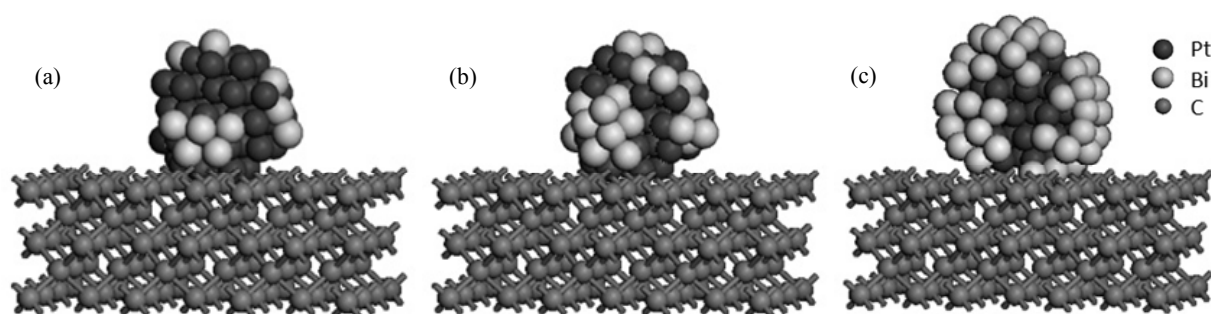
Catalyst	Particle size (nm)		Conversion <sup>b</sup> (%)	Yield of DIHA (%)	Selectivity for products (%)					
	XRD <sub>Pt</sub> <sup>a</sup>	TEM <sub>Pt-Bi</sub>			DIHA	GLYA	GLYCA	GLYHD	OXALA	C <sub>1</sub> <sup>b</sup>
Pt/C	3.4	3.1	70.3	6.5	9.2	66.4	8.0	1.3	0.0	15.1
Bi/C	—	—	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5%Pt-3%Bi/C	2.9	3.7	74.4	34.6	46.5	10.2	6.6	0.0	19.5	17.2
5%Pt-5%Bi/C	<1.0	3.8	91.5	44.8	49.0	5.4	0.0	0.0	39.9	5.7
5%Pt-7%Bi/C	<1.0	7.1	72.0	27.9	38.8	15.6	14.1	0.0	18.6	12.8

Reaction conditions: catalyst 0.5 g, aqueous solution of glycerol 50 ml (0.1 g/ml), 60 °C, oxygen 150 ml/min, 6 h.

<sup>a</sup>Mean particle size of platinum as measured by XRD and according to the Scherrer-Warren equation.

<sup>b</sup>Mono-carbon products (mainly CO<sub>x</sub> and formaldehyde).

GLYA: glyceric acid; GLYCA: glycolic acid; GLYHD: glyceraldehydes; OXALA: oxalic acid.

**Fig. 5.** Bi blocked Pt particles in 5%Pt-3%Bi/C (a), 5%Pt-5%Bi/C (b), and 5%Pt-7%Bi/C (c).

(Table 1). On the surface of the monometallic Pt catalyst, mainly free glyceric acid (GLYA, with 66.4% selectivity) was detected and the selectivity for DIHA was low (9.2%, Table 1). These results are consistent with those reported in the literature [9,12,22–25]. It is interesting to find that the selectivity for DIHA reached 46.5% on 5%Pt-3%Bi/C and it then increased to 49.0% on 5%Pt-5%Bi/C. However, it decreased to 38.8% on 5%Pt-7%Bi/C. Meanwhile, the conversion of glycerol changed with the same trend over these catalysts (Table 1), which increased from 74.4% on 5%Pt-3%Bi/C to 91.5% on 5%Pt-5%Bi/C and then decreased to 72.0% on 5%Pt-7%Bi/C. This higher DIHA selectivity of the Pt-Bi bimetallic catalysts compared with that of single Pt can be attributed to the site-blocking function of Bi atoms on Pt(111) and a Bi sub-monolayer on Pt would increase the formation of DIHA in glycerol [12]. At the same time, oppositely changed GLYA selectivity may also account for the higher DIHA selectivity of 5%Pt-5%Bi/C as

GLYA was found to selectively block the active sites that are predominantly responsible for DIHA formation [18]. The volcanic trend can be attributed to that a lower amount of Bi increased the dispersion of Pt but a higher amount of Bi led to a severe wrapping of Pt particles, which blocked access to Pt (Fig. 5).

The stability of a 5 times recycled 5%Pt-5%Bi/C catalyst is summarized in Table 2. We found that the product distribution remained unchanged during these cycles while the conversion of glycerol decreased slightly from 91.5% to 84.8%.

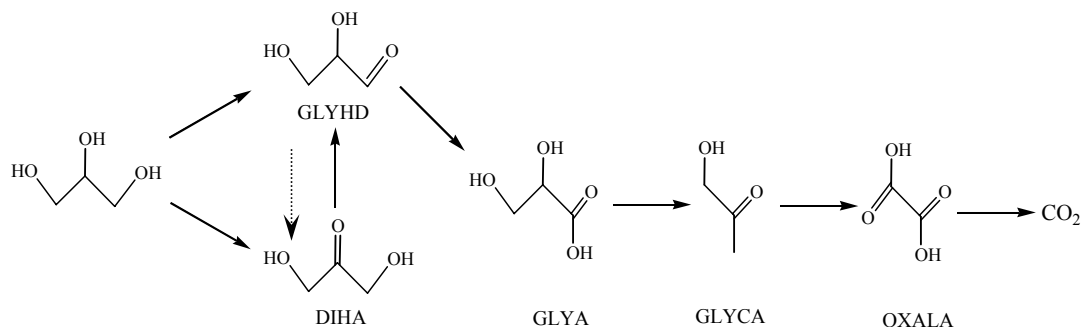
The time course of the product selectivity of the reactions over bimetallic Pt-Bi catalysts with different Bi content is compared in Fig. 6. At 30 min, the selectivity for DIHA over 5%Pt-5%Bi/C reached 94.8% (Fig. 6(a)) but it dropped continuously to 57.0% at 120 min and then decreased slightly to 49.0% at the end of the reaction (360 min). The selectivity for GLYA changed in a similar trend to that of

**Table 2** Recycling of the 5%Pt-5%Bi/C catalyst<sup>\*</sup>

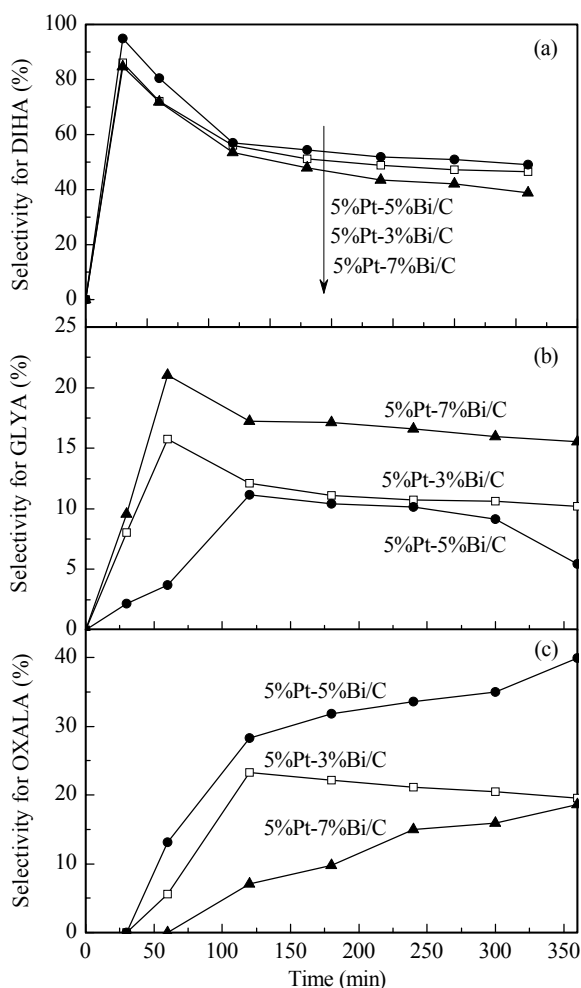
Time	Conversion (%)	Yield of DIHA (%)	Selectivity for products (%)					
			DIHA	GLYA	GLYCA	GLYHD	OXALA	C <sub>1</sub>
1	91.5	44.8	49.0	5.4	0.0	0.0	39.9	5.7
2	88.7	43.0	48.5	5.3	0.0	0.0	40.8	5.4
3	86.8	41.3	47.6	6.0	0.0	0.0	39.4	7.0
4	85.4	41.0	48.0	5.6	0.0	0.0	40.4	6.0
5	84.8	40.5	47.7	5.4	0.0	0.0	40.0	6.9

<sup>\*</sup>The used catalyst was recovered simply by filtration and washed with deionized water before the next cycle.

Reaction conditions: catalyst 0.5 g, aqueous solution of glycerol 50 ml (0.1 g/ml), 60 °C, oxygen 150 ml/min, 6 h.



**Scheme 1.** Reaction scheme for the selective oxidation of glycerol.



**Fig. 6.** Selectivity for DIHA (a), GLYA (b), and OXALA (c) versus the reaction time. Reaction conditions: catalyst 0.5 g, aqueous solution of glycerol 50 ml (0.1 g/ml), 60 °C, oxygen 150 ml/min.

DIHA but it reached a maximum at 120 min (Fig. 6(b)) and then decreased continuously afterwards. The sharp decrease in the selectivity for DIHA in the initial period (first 30 min, as shown in Fig. 6(a)) indicated that DIHA is the primary intermediate in glycerol oxidation. Additionally, the absence of a detectable amount of GLYHD (Scheme 1) is not surprising since it could easily be oxidized to GLYA after for-

mation by the conversion of DIHA [25–27]. The continuously decreased selectivity for GLYA after 130 min of the reaction is because it was further oxidized to other products among which OXALA was the major product with the continuous increase in selectivity (Fig. 6(c), Scheme 1).

The same product selectivity trend in the glycerol oxidation was detected over 5%Pt-3%Bi/C and 5%Pt-7%Bi/C (Fig. 6). However, the selectivity for GLYA over 5%Pt-5%Bi/C was lower than that of 5%Pt-3%Bi/C and 5%Pt-7%Bi/C throughout the whole time course.

On the basis of these product distributions, we think that the reaction scheme of the selective oxidation of glycerol over the bimetallic Pt-Bi catalyst is as illustrated in Scheme 1. On the surface of Pt-Bi, glycerol was initially oxidized to DIHA and the formed DIHA was further oxidized to GLYA (via GLYHD), OXALA, or CO<sub>2</sub>.

### 3 Conclusions

The performance of the monometallic Pt/C catalyst for glycerol oxidation to DIHA was improved greatly because of the presence of Bi. 5%Pt-5%Bi/C was more selective for the production of DIHA from glycerol and the selectivity for DIHA reached 49.0% at a 91.5% conversion of glycerol. This superior performance may be attributed to the presence of specially configured Pt-Bi nanoparticles, which are highly dispersed (3.8 nm) on the active carbon support.

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## 2010 年国内化学类期刊影响因子和总被引频次排序表

(中国科学技术信息研究所 2011 年 12 月公布)

期刊名称	影响因子		总被引频次		期刊名称	影响因子		总被引频次	
	数值	排名	数值	排名		数值	排名	数值	排名
色谱	1.926	1	2035	6	应用化学	0.524	19	1532	11
分子催化	1.729	2	828	21	煤炭转化	0.464	20	579	25
分析化学	1.529	3	4352	1	中国科学化学	0.442	21	979	18
分析试验室	1.096	4	1905	8	Chinese Journal of Structural Chemistry	0.440	22	623	23
分析测试学报	1.080	5	1961	7	化学分析计量	0.426	23	386	28
物理化学学报	0.920	6	2265	4	化学研究与应用	0.411	24	998	16
高等学校化学学报	0.839	7	3932	2	影像科学与光化学	0.368	25	173	34
环境化学	0.825	8	1567	10	化学通报	0.342	26	982	17
化学学报	0.801	9	3025	3	Chinese Chemical Letters	0.328	27	699	22
<b>催化学报</b>	0.777	10	<b>1713</b>	<b>9</b>	化学与生物工程	0.319	28	563	26
无机化学学报	0.764	11	2039	5	电化学	0.303	29	232	33
化学进展	0.743	12	1499	12	化学研究	0.282	30	263	32
燃料化学学报	0.733	13	1069	15	Chemical Research in Chinese Universities	0.272	31	270	31
高分子学报	0.725	14	1452	13	化学与黏合	0.259	32	280	29
高分子通报	0.717	15	943	19	化学试剂	0.238	33	617	24
分子科学学报	0.640	16	277	30	Chinese Journal of Polymer Science	0.224	34	162	35
分析科学学报	0.637	17	943	19	合成化学	0.217	35	400	27
有机化学	0.623	18	1402	14					