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Synthesis of Molybdenum Disulfide from Waste Mo Materials†

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MoS_2 was synthesized from waste Mo materials and Na_2S by a chemical method. The effect of reaction conditions on the yield of MoS_2 and the recovery rate of Mo was investigated. The results showed the yield of MoS_2 and the recovery rate of Mo was remarkably influenced by the leaching and extraction conditions, including time, temperature and ratio of liquid to solid. The as-prepared MoS_2 comprises clusters of nanoparticles with *ca.* 100 nm sizes. This work exhibits potential applications in the recovery of waste Mo materials.

Key Words: Molybdenum disulfide, Waste Mo materials, Recovery.

INTRODUCTION

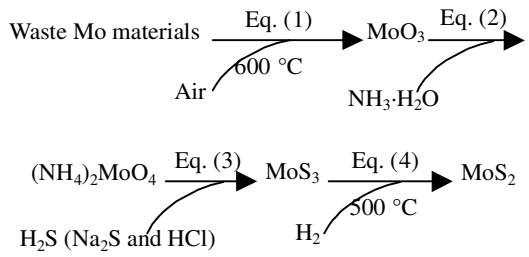
Molybdenum (Mo), a rare metal element, is widely applied in petrochemical industry, machinery industry, iron and steel industry and electron industry. The recovery of Mo from waste Mo materials is a significant work that has attracted considerable attention[1,2]. Molybdenum disulfide (MoS_2) is a typical layered inorganic compound. MoS_2 is often used as solid lubricants to reduce contact wear of machines. Moreover, MoS_2 also presents excellent catalytic properties[3,4] and has been widely used as hydrodesulfurization catalysts in petrochemical industry[5]. The laminar structure of MoS_2 is generally regarded as a crucial reason for its excellent properties in catalysis and lubrication. Several new chemical methods have been recently developed to synthesize MoS_2[6,7]. The present work studied the synthesis of MoS_2 from waste Mo materials with chemical techniques.

EXPERIMENTAL

All chemical reagents used were of analytical grade. Waste Mo materials purchased was analyzed using a chemical method, the results of which showed the Mo per cent was 19.4 %. The obtained product was characterized using a Rigaku model D/Max-γB X-ray diffractometer (XRD) and a Hitachi model H-800 transmission electron microscopy (TEM), respectively. The recovery process is provided in Fig. 1.

RESULTS AND DISCUSSION

Formation of MoS_2 from waste Mo materials: The transformation process of waste Mo materials to MoS_2 is shown in eqns. 1-4.



$$\text{Waste Mo materials} + O_2 \xrightarrow{600^\circ C} MoO_3 \quad (1)$$

$$MoO_3 + 2NH_3\cdot H_2O \rightarrow (NH_4)_2MoO_4 + H_2O \quad (2)$$

$$3H_2S + (NH_4)_2MoO_4 + 2HCl \rightarrow MoS_3 + 4H_2O + 2NH_4Cl \quad (3)$$

$$MoS_3 + H_2 \xrightarrow{500^\circ C} MoS_2 + H_2S \quad (4)$$

Effect of recovery conditions: The leaching conditions presented remarkable effects on the yield of MoS_2 and the recovery rate of Mo. The recovery rate increased with the prolonged leaching time (Fig. 2a), which can be explained using the relation between reaction time and yield of resultants. A high temperature remarkably increased the volatilization of $NH_3\cdot H_2O$. Thus, the recovery rate decreased with the increasing reaction temperature (Fig. 2b). Generally, an increase in the amount of reactants may lead to high yields of resultants. The amount of $NH_3\cdot H_2O$ was increased when the ratio of liquid to solid was magnified. Thus, the recovery rate increased with the increasing ratio of liquid to solid (Fig. 2c).

Figs. 2d, e and f provide the influence of extraction conditions on the yield of MoS_2 and the recovery rate of Mo, including extraction time, extraction temperature and ratio of methyl alcohol to filter liquor. As shown the three figures, the

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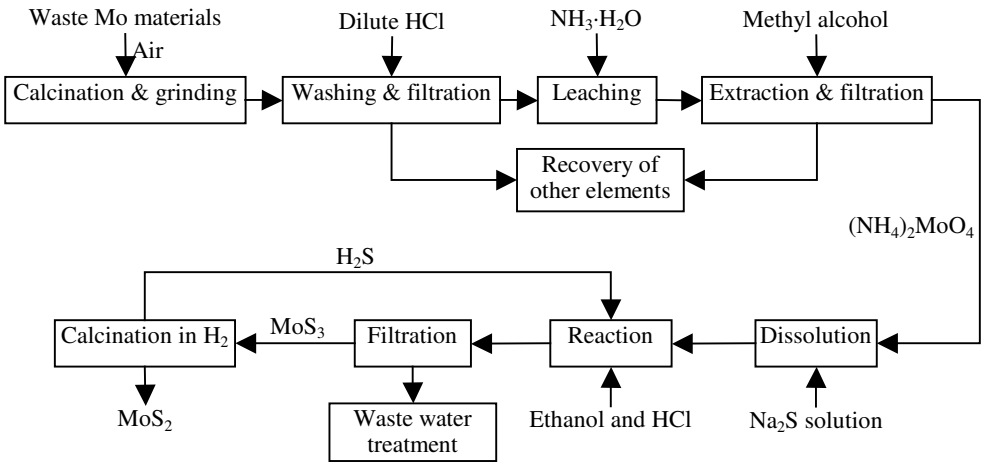


Fig. 1. synthesizing process of MoS_2 from waste Mo materials

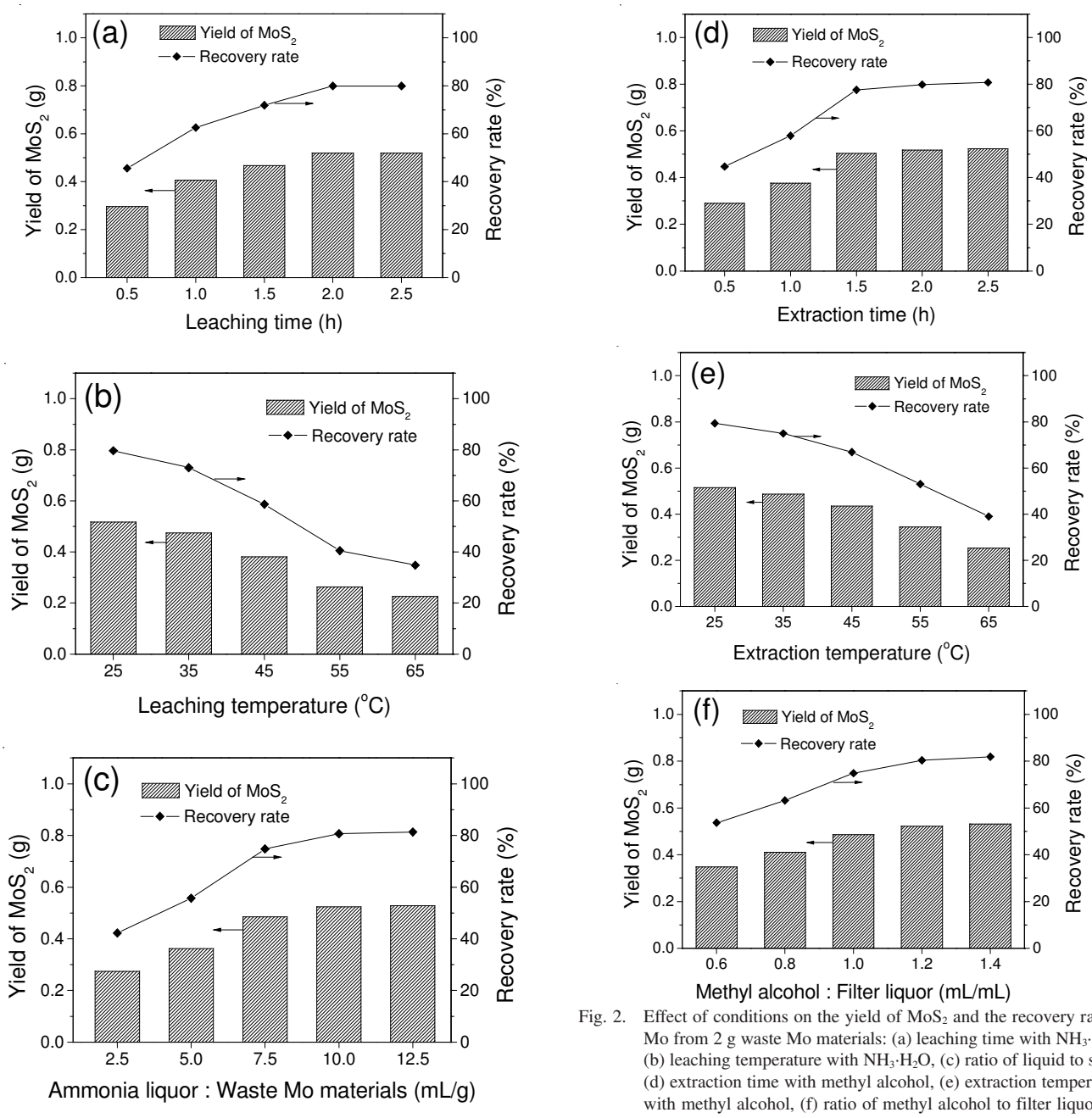
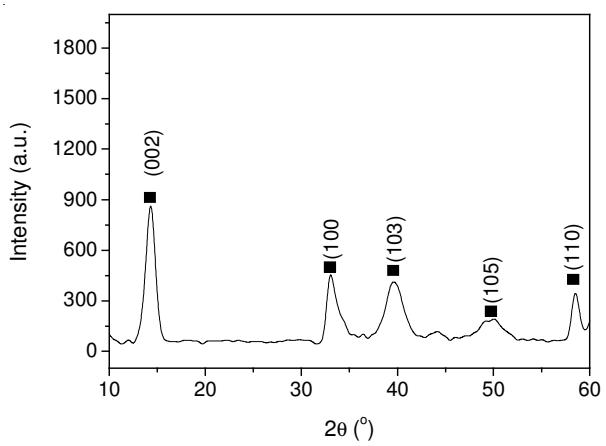


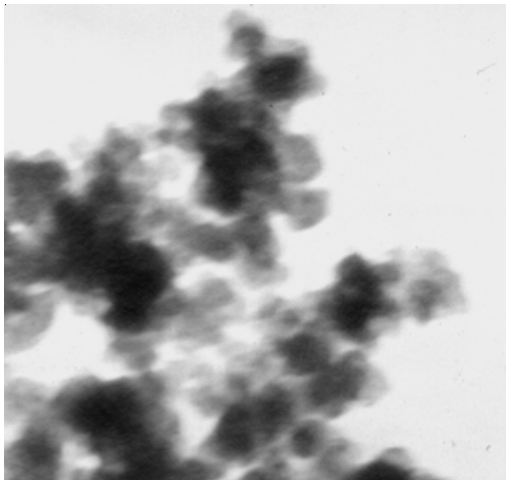
Fig. 2. Effect of conditions on the yield of MoS_2 and the recovery rate of Mo from 2 g waste Mo materials: (a) leaching time with $NH_3 \cdot H_2O$, (b) leaching temperature with $NH_3 \cdot H_2O$, (c) ratio of liquid to solid, (d) extraction time with methyl alcohol, (e) extraction temperature with methyl alcohol, (f) ratio of methyl alcohol to filter liquor

extraction conditions with methyl alcohol affected the yield and the recovery rate in a similar way to the leaching conditions with $NH_3 \cdot H_2O$.

Characterization: Fig. 3a provides the XRD pattern of the product from the waste Mo materials. According to the PDF 37-1492 card and a previous literature[6], the main diffraction peaks of the XRD pattern were indexed to (002), (100), (103), (105) and (110) of MoS_2, respectively. This indicates that MoS_2 was successfully synthesized from waste Mo materials. As shown in Fig. 3b, the as-prepared MoS_2 comprises clusters of nanoparticles with *ca.* 100 nm sizes.



(a)



(b)

Fig. 3. Results of characterization: (a) XRD and (b) TEM

Conclusion

MoS_2 can be synthesized from waste Mo materials. The leaching and extraction conditions remarkably affect the yield of MoS_2 and the recovery rate of Mo. The as-prepared MoS_2 comprises clusters of nanoparticles with *ca.* 100 nm sizes.

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