# Technology Section

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## PREPARATION OF ZINC SULPHATE FROM ZINC DROSS

Arjamand Khan, M. Alauddin, Bibi Hajra and M.A. Khattak

*PCSIR Laboratories, Peshawar*

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Production of zinc sulphate from zinc dross was studied by a procedure previously worked out for preparation of metallic sulphates from sulphides. The reaction between zinc dross and ammonium sulphate in reducing atmosphere gives zinc sulphate and vaporous products i.e. sulphur dioxide, ammonia and water. The effects of temperature, ammonium sulphate and time were studied. The above reaction may be utilised for large scale production of zinc sulphate from zinc dross.

*Keywords:* Zinc sulphate, Zinc dross, Ammonium sulphate

### INTRODUCTION

The reduction of stibnite by methane into metallic antimony and chiefly hydrogen sulphide was reported by Khundkar and Eusuf [1]. Ammonium sulphate reacts with metal sulphides in a stream of reducing gases to form metal sulphates [2]. The reaction between ammonium sulphate and metallic sulphides starts at a temperature of 150°-200°C with a slow fusion and decomposition of ammonium sulphate to ammonium bisulphate and ammonia as represented by the equation:-

$$(NH_4)_2SO_4 \longrightarrow NH_4HSO_4 + NH_3$$

With the rise of temperature ammonium bisulphate starts reacting with zinc dross producing an anhydrous zinc sulphate and vaporous products i.e. $SO_2$, $NH_3$ and $H_2O$ according to the following reaction:-

$$2NH_4HSO_4 + \text{Zn dross} \longrightarrow ZnSO_4 + SO_2 + 2NH_3 + 2H_2O$$

The preparation of metallic sulphates from sulphides in reducing atmosphere was studied in these laboratories [3].

From a consideration of the relative free energy changes with temperature, the reduction of sulphides by hydrogen was first suggested by Ellingham [4]. Clarke and Spittle [5] studied the reaction between Hydrogen and Iron pyrites. According to the free energy of formation [6], methane should be thermodynamically unstable above 500°C. The pyrolysis of methane is complex, it is known [7] to take place mainly as:

$$CH_4 \longrightarrow C + 2H_2$$

Optimum condition for the highest conversion of zinc sulphate from zinc dross by studying three parameters namely effect of temperature, ammonium sulphate and time was established. The present investigation provides us with an improved method of preparing zinc sulphate by mixing powdered zinc dross with ammonium sulphate in reducing atmosphere.

### EXPERIMENTAL

Zinc dross was thoroughly washed with water, dried and then ground into fine powder (–100 mesh). The effects of temperature, ammonium sulphate and time were determined in a bench-scale experiments designed in these Laboratories. The essential features of the diagram are shown in Figure 1. The method comprises of heating zinc

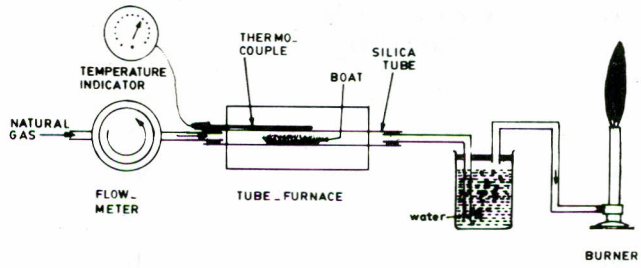


Fig. 1. Apparatus for the production of zinc sulphate from zinc dross.

dross powder with fixed and/or different quantities of ammonium sulphate. The silica tube (dia 3 cm) was introduced in a tube furnace. The mixture of zinc dross and ammonium sulphate was placed in a porcelain boat. The boat was placed in a leak-proof atmosphere of sui gas inside the silica tube at the central zone of the electric tube furnace. The chromle/alumle thermocouple was connected with temperature indicator and the temperature thus measured. On one side the silica tube was connected to a flow meter and on other to glass cylinder containing water and then to a gas burner. The sui gas was then passed through flow meter for a definite period and the flue gases were bubbled in water. The rate of flow of gas was approximately 200 bubbles per minute which comes to about

140 ml. per minute. The material in the boat was analysed to determine percentage conversion/recovery of zinc sulphate.

The effect of temperature from 150-400° with an interval of 50° was determined, the percentage yield was found out as shown in Table 1. It was observed that at a temperature of 300°, the recovery was 100% by keeping the time of reaction constant i.e., one hour in all the experiments.

In Table 2 it is seen that by varying the ratio of zinc dross: $(NH_4)_2SO_4$ the percentage coversion of zinc dross to zinc sulphate was determined at a temperature of 300°C. The time of reaction was kept constant at one hour in the usual way. The maximum conversion (100%) takes place when the ratio of zinc dross: Ammonium sulphate was 1:2 and remains constant when it was increased to 1:3.

The results indicated in Table 3 shows that at a temperature of reaction of 300° for 25-30 minutes maximum recovery was obtained. It was noted that by keeping the temperature and quantity of ammonium sulphate constant the percentage yield was found to be 100% between 25 and 30 min.

*Estimation of zinc.* Zinc has been determined by a standard method [8].

## RESULTS AND DISCUSSIONS

Table 1-3 reveals the average percentage yield values of zinc sulphate formed from the reaction between zinc dross and ammonium sulphate. Figs 2-4 indicate the relationship of the effects of temperature, ammonium sulphate and time against the percentage conversion of zinc sulphate.

The work was undertaken with a view to study the effects of temperature, ammonium sulphate and time for maximum conversion of zinc dross to zinc sulphate. By employing these three factors 100% conversion of zinc dross to zinc sulphate was obtained.

Table 1. Effect of temperature. Time of reaction = One hour.

| S.No. | Quantity ingredients | Temperature of reaction °C | Weight of product g. | Weight of Zn estimated in $ZnSO_4$ solution g. | Percentage conversion to $ZnSO_4$ |
|---|---|---|---|---|---|
| 1. | 1g. Zn dross + 3g. $(NH_4)_2SO_4$ | 150 | 3.7819 | 0.0004 | Less than 0.04 |
| 2. | " | 200 | 3.6338 | 0.455 | 45.5 |
| 3. | " | 250 | 2.8908 | 0.648 | 64.8 |
| 4. | " | 300 | 2.9534 | 1.02 | 100 |
| 5. | " | 350 | 2.8382 | 0.994 | 100 |
| 6. | " | 400 | 2.5376 | 1.01 | 100 |

Table 2. Effect of ammonium sulphate. Time of reaction = One hour.
Temperature of reaction = 300°C

| S.No. | Quantity of ingredients | Weight of product g. | Weight of Zn estimated in $ZnSO_4$ solution g. | Percentage conversion to $ZnSO_4$ |
|---|---|---|---|---|
| 1. | Ig. Zn dross + 1g. $(NH_4)_2SO_4$ | 1.5666 | 0.702 | 70.2 |
| 2. | " " " + 1.50 " " | 1.9205 | 0.864 | 86.4 |
| 3. | " " " +1.75 " " | 2.1512 | 0.918 | 91.8 |
| 4. | " " " +2 " " | 2.2258 | 1.00 | 100 |
| 5. | " " " +2.5 " " | 2.5023 | 1.00 | 100 |
| 4. | " " " +3 " " | 2.9534 | 1.00 | 100 |

Table 3. Effect of time. Temperature of reaction = 300°C.

| S. No. | Quantity of ingredients | Time of reaction minutes | Weight of product g. | Weight of Zn estimated in $ZnSO_4$ solution | Percentage conversion to $ZnSO_4$ |
|---|---|---|---|---|---|
| 1. | 1g. Zn dross + 2g. $(NH_4)_2SO_4$ | 5 | 2.4804 | 0.8960 | 89.60 |
| 2. | | 10 | 2.3488 | 0.9292 | 92.92 |
| 3. | " | 15 | 2.4372 | 0.9579 | 95.79 |
| 4. | " | 20 | 2.3067 | 0.9792 | 97.92 |
| 5. | " | 25 | 2.3522 | 0.9987 | 100 |
| 6. | " | 30 | 2.3607 | 1.010 | 100 |

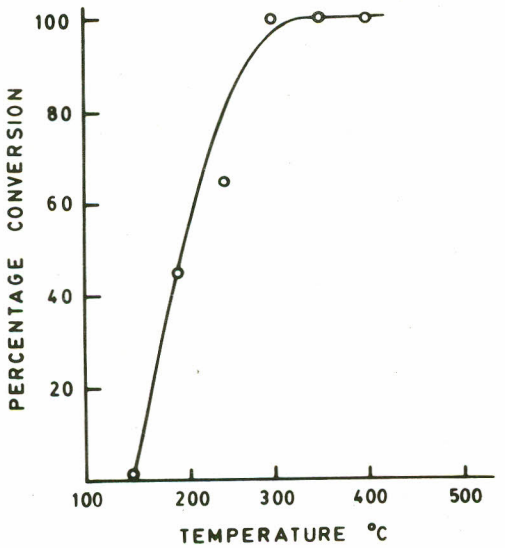


Fig. 2. Reaction of Zn dross with $(NH_4)_2SO_4$ at different temperature.

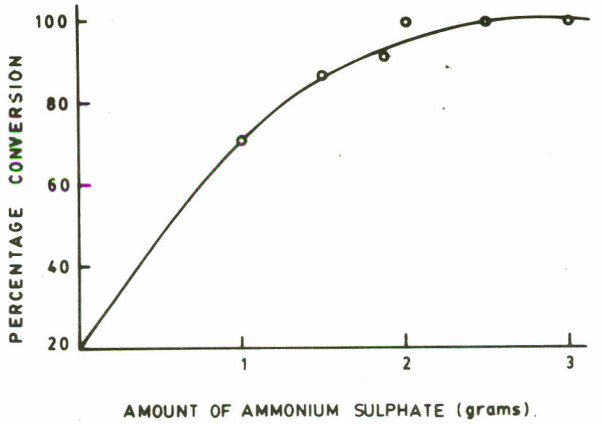


Fig. 3. Reaction of Zn dross with different amount of $(NH_4)_2SO_4$.

The percentage of conversion at 150° is only less than 0.04% as can be seen from Table 1. At 200° there is a high rise to 45.5%. But at 300° the rate of conversion reaches to 100% by keeping the ratio of zinc dross to ammonium sulphate to 1:3. It is clear from Table 2 that the optimum ratio between zinc dross and ammonium sulphate is 1:2. The time required for the reaction ranges from 25 minutes to 30 minutes, as obvious from Table 3.

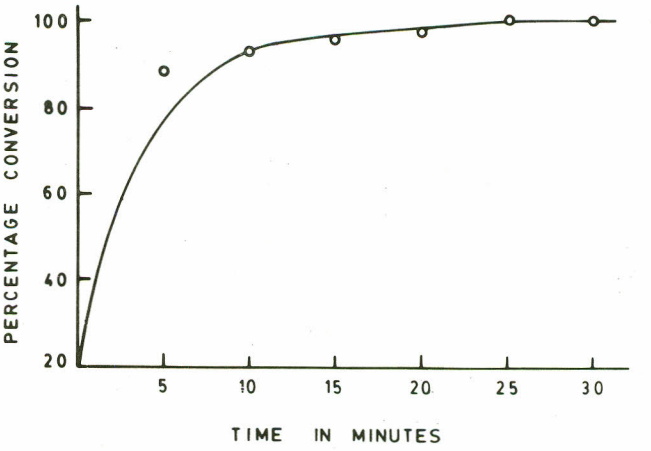


Fig. 4. Reaction of Zn dross with $(NH_4)_2SO_4$ at different interval time.

It is seen from the three above tables that by taking zinc dross and ammonium sulphate in the ratio 1:2 and heating for a period of 25 minutes the maximum conversion to 100% at 300° is achieved.

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