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SHORT COMMUNICATION

Preparation of High Purity Amorphous Boron Powder

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ABSTRACT

Amorphous boron powder of high purity (92-94 %) with a particle size of 1-2 mm is preferred as a fuel for fuel-rich propellants for integrated rocket ramjets and for igniter formulations. This paper describes the studies on process optimisation of two processes, ie, oxidative roasting of boron (roasting boron in air) and roasting boron with zinc in an inert medium for preparing high purity boron. Experimental studies reveal that roasting boron with zinc at optimised process conditions yields boron of purity more than 93 per cent, whereas oxidative roasting method yields boron of purity ~ 92 per cent. Oxidative roasting has comparative edge over the other processes owing to its ease of scale-up and simplicity

Keywords: Amorphous boron powder, igniter formulations, process optimisation, preparation, high purity, boron, fuel-rich propellent, oxidative roasting

1. INTRODUCTION

Amorphous boron powder (ABP) has comparative edge over the conventional fuels like aluminum, magnesium, etc. owing to its high heat of combustion[1] for use in fuel-rich propellants for integrated rocket ramjets. The low atomic weight, high heat output, ready ignitability with KNO_3, releasing large amount of heat and persistent burning even at low-pressure[2] make boron-based pyrotechnic composition a very attractive igniter composition. High Energy Materials Research Laboratory, Pune, has established a process for the preparation of ABP Gr II of 85-88 per cent purity. High purity amorphous boron powder Gr I of 92-94 per cent purity is preferred for use in fuel-rich propellants for integrated rocket ramjets and igniter formulations for better performance. Main impurity in ABP Gr II is magnesium in the form of magnesium polyborides. Various methods reported[3,4] for improving the purity of ABP Gr II to ABP Gr I are:

(a) Roasting boron with zinc in an inert medium

(b) Oxidative roasting of boron in air

(c) Treating boron with alkali metal fluoroborates

(d) Extraction of boron with fused boric oxide.

However, methods (a) and (b) are considered as promising routes for improving the purity of amorphous boron. In method (a), ABP Gr II is roasted with zinc in an inert atmosphere of argon at higher temperature to improve the purity of boron. The reaction involved is:

$$MgB_x + 2Zn \overset{\text{(Inertmedium)}}{\rightleftharpoons} MgZn_2 + xB$$

where x = 2, 4, 6, 12.

Oxidative roasting of boron involves roasting of ABP Gr II in air at higher temperature to improve

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Table 1. Data on time of roasting of ABP Gr II with zinc in an inert medium

Time of roasting (min)	Purity of ABP Gr II		Purity of ABP Gr I		Yield (%)
	B (%)	Mg (%)	B (%)	Mg (%)	
15	86	10.1	88.8	5.3	80
30	86	10.1	93.5	3.9	85
45	86	10.1	93.2	5.6	80
60	86	10.1	93.0	4.5	79

Temperature of roasting: 600 °C, batch size :10 g

the purity. During roasting, oxidation of magnesium from magnesium poyborides is more rapid than the oxidation of boron[3].

The probable reactions are:

$$2MgB_x + O_2 \longrightarrow 2MgO + 2xB$$

where x = 6,12.

$$4B + 3O_2 \longrightarrow 2B_2O_3$$

The objective of this study is to optimise the process parameters (time of roasting, temperature of roasting, etc) for roasting of boron with zinc in an inert medium and oxidative roasting of boron at bench scale. Scale-up of the bench scale method is also investigated. A comparative study of both the processes has also been carried out.

2. EXPERIMENTAL PROCEDURE

2.1 Raw materials

(i) Amorphous boron powder (ABP) Gr II conforming to HEMRL/CEPP/PS/3 54

(ii) Zinc powder (GR Grade) procured form Loba Chemie.

2.2 Equipment

Muffle furnace (capacity) (i) 3 kW (ii) 22 kW

2.3 Procedure

2.3.1 *Roasting ABP Gr II with Zinc in Inert Medium*

Bench-scale studies were undertaken to optimise various process parameters for roasting ABP Gr II with zinc in an inert medium. A homogeneous mixture of ABP Gr II and zinc powder (10 g each) was spread as a thin layer in a SS tray and was subjected to roasting in an inert medium of argon in a muffle furnace. The roasted boron was then leached with hydrochloric acid to remove the soluble impurities. It was further washed with ethanol/methanol mixture, filtered and dried to get pure boron. Effect of temperature and time of roasting were studied to achieve the optimised parameters for better yield and purity. The results have been summarised in Tables 1 and 2. The bench-scale process was

Table 2. Data on temperature of roasting of ABP Gr II with zinc in an inert medium

Temperature of roasting (°C)	Purity of ABP Gr II		Purity of ABP Gr I		Yield (%)
	B (%)	Mg (%)	B (%)	Mg (%)	
500	86	10.1	90.6	6.3	77
550	86	10.1	93.0	3.5	78
600	86	10.1	93.5	2.3	85
650	86	10.1	93.0	3.0	66
700	86	10.1	75.6	5.8	44

Time of roasting : 30 min, batch size : 10 g subsequently scaled-up to 100 g batch size using a muffle furnace of larger capacity (22 kW) with the optimised process parameters.

2.3.2 Oxidative Roasting

Bench-scale studies were undertaken to optimise various process parameters like temperature and time of roasting of ABP Gr II. ABP Gr II (10 g) was spread as a thin layer in an SS tray and was subjected to roasting in a muffle furnace. The roasted boron was leached with hydrochloric acid, washed with ethanol/ methanol mixture, filtered and dried to get pure boron. Effect of temperature and time of roasting were studied to achieve the optimised parameters for better yield and purity. The results of the above experiments are summarised in Tables 3 and 4. With the optimised process parameters at bench scale, the process was further scaled-up to 300 g level using a muffle furnace of larger capacity (22 kW).

3. RESULTS & DISCUSSION

The purity of ABP Gr I can be achieved by roasting ABP Gr II with zinc in an inert medium or by roasting ABP Gr II in air. In both the methods, time and temperature of roasting play a vital role in obtaining the required purity of ABP Gr I.

From Table 1 it is evident that the highest purity (93.5 %) and yield (85 %) can be achieved by roasting ABP Gr II with zinc in an inert medium for 30 min. Beyond 30 min of roasting, no further improvement on purity/yield was obtained. Hence, the time of roasting was optimised as 30 min.

Data on temperature of roasting of ABP Gr II with zinc in inert medium as reflected in Table 2 indicates that the highest purity (93.5%) can be obtained at a roasting temperature of 600 °C. With further increase in temperature, a decreasing trend in purity was observed. This is presumably due to the predominance of backward reaction[3] (ie $MgZn_2 + xB \rightarrow MgB_x + 2Zn$) above 600 °C.

It is thus concluded that to achieve the maximum purity and yield by roasting ABP Gr II with zinc in an inert medium, the ideal roasting temperature and time are 600 °C and 30 min. The same temperature and time have been found to be ideal for oxidative roasting of ABP Gr II also.

It was observed that layer thickness of the charge is also one of the critical factors that determine the'purity of the final product. Experimental studies revealed that for the method of roasting ABP Gr II with zinc in an inert medium, the layer thickness

Table 3. Data on time of roasting ABP Gr II in air

Time of roasting (min)	Purity of ABP Gr II		Purity of ABP Gr I		Yield (%)
	B(%)	Mg (%)	B (%)	Mg (%)	
20	86.2	9.54	92.27	2.28	70
30	86.2	9.54	92.52	2.37	80
40	86.2	9.54	89.45	1.96	75

Temperature of roasting 600 °C, batch size 10 g.

Table 4. Data on temperature of roasting of ABP Gr II in air

Temperature of roasting (°C)	Purity of ABP Gr II		Purity of ABP Gr I		Yield (%)
	B (%)	Mg (%)	B(%)	Mg (%)	
525	86.2	9.54	90.14	4.90	80
550	86.2	9.54	91.92	3.39	76
575	86.2	9.54	91.90	2.36	80
600	86.2	9.54	92.36	1.69	81
625	86.2	9.54	91.1	1.8	56

Time of roasting 30 min, batch size 10 g.

of the charge material is to be maintained between 1.3 mm and 1.6 mm. However for oxidative roasting method, a layer thickness up to 3.8 mm would give the desired purity. Hence in oxidative roasting method, more quantity of ABP Gr II can be upgraded to ABP Gr I per batch.

The process of roasting of ABP Gr II with zinc in an inert medium could be scaled-up to 100 g level with the optimised process parameters. The maximum purity of ABP obtained was 92-94 per cent and yield was 75-80 per cent. However, the process of oxidative roasting of boron could be scaled-up to 300 g level with the optimised process parameters using the same muffle furnace. The purity of boron obtained was 91-93 per cent with yield of about 70-80 per cent.

It was observed that the scale-up of process of oxidative roasting of ABP Gr II was relatively easiêr and cheaper than the process of roasting ABP Gr II with zinc in an inert medium. Moreover, in roasting ABP Gr II with zinc, zinc is always present as an added impurity in the form of zinc borides. Bunin[3] has reported that during roasting of ABP Gr II with zinc in an inert medium, zinc is incorporated in boron lattice and its content in boron may reach 18-20 mass per cent. The chemical formula of zinc boride was established as $B_{22}\,Zn$. Thermal treatment in high vacuum at 1000 °C, results in the dissociation of zinc boride with the evolution of zinc. After thermal treatment, 0.2 per cent zinc remains in ABP Gr II. Hence by adopting the process of roasting ABP Gr II with zinc, an additional thermal treatment step would be required to obtain pure boron. Boron reacts with most metals at high temperatures and selection of suitable material of construction for the reaction chamber would also be difficult. Also, use of inert medium makes the process of roasting ABP Gr II with zinc more costly and cumbersome. Considering the above aspects, the method of oxidative roasting of ABP Gr II has comparative edge over the method of roasting of ABP Gr II with zinc in an inert medium.

4. CONCLUSION

Roasting boron with zinc in the ratio 1:1 at 600 °C for 30 min in an inert atmosphere of argon could yield ABP Gr I having a purity of 93-94 per cent, whereas by oxidative roasting method, boron of up to 92 per cent purity could be obtained. However, scaling-up of the process of oxidative roasting has been found to be relatively easier and has a comparative edge over the method of roasting boron with zinc.

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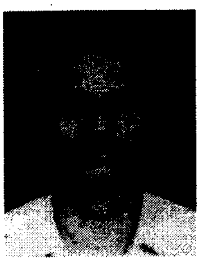

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