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STUDY OF SOLUTION COMBUSTION PROCESS FOR PRODUCTION OF METAL OXIDES AND ANALYSIS OF FUEL EFFICIENCY PARAMETERS

ROHIT KUMAR*, AMIT SHARMA, SANDEEP SALARIA
Arni School of Technology, Arni University, Kathgarh, Indora
*E-mail:bawarohit1992@gmail.com

ABSTRACT

Solution combustion process is a well-known method for production of metal oxides. Different metal oxides have been produced so for using this economical and simple method. The parameters that led it to be area of research interest are efficiency of different fuels, fuel to oxidizer ratio, mixed fuel approach, feasibility of process towards production of different metal oxides. Here mixed fuel with different combinations of fuel to oxidizer ratio and also of two fuels is used. Three samples were prepared and characterized by flame observation. XRD, and SEM techniques. Fuel to oxidizer ratio effects thermionic property of product material and thus affects properties such as crystallinity, particles size and hardness etc. of as produced metal oxide. Alumina powder was undertaken as objective of study. Results analyzed are in agreement with literature data.

Keywords: Metal oxide, Alumina, SHS, Solution combustion, fuel Oxidizer, ceramic oxides, nano oxides.

1. INTRODUCTION

Metal oxides are well known for their numerous applications. Metal oxides find their use in manufacturing industry such as tools and parts fabrication. Some most common applications include spark plugs, tap washers, pump seals, grinding media, abrasion resistant tiles, cutting tools, and wear parts for the textile and paper industries. Researchers from all over the world are working with different methods to produce metal oxides. Commonly used methods for production of metal oxides are sol-gel method, metal matrix method, SHS i.e. self propagating high temperature synthesis or solution combustion [1-5]. Solution combustion or Combustion synthesis (CS) is a excellent, economic and easy mechanism for production of metal oxides. In last few years, a number of new breakthroughs in this field have been observed, notably for production of new catalysts and nano carriers with efficiency better than those for same traditional materials. Alumina (Aluminium oxide) is a widely used metal oxide has many applications in industry (i.e. Abrasive, wear resistant, coating industry etc.) [6-10].SHS technology is utilizing less expensive chemical energy instead of the electrical power, thus is simple to perform production mechanism [11-15]. Layer-by-layer heat release that ensures the feasibility to operate with large amount of substance and feasibility of setting up the in-line production [16-25]. Many articles reported synthesis of alumina by SHS (solution combustion) process. Parameter that is most commonly reported in most of papers is effect of fuel to oxidizer ratio on synthesis of metal oxide and properties of product formed [6, 7, 8, 26-40]. Glycine, ammonium acetate, ammonium tartarate, urea have been explored as fuels. Different combinations of fuel to oxidizer ratio have been analysed [5-10][41] . Here in this paper mixed fuel with different combinations of fuel to oxidizer ratio and also of two fuels is used. Three samples were prepared and characterized by flame observation. XRD, and SEM techniques. Fuel to oxidizer ratio effects thermionic property of product material and thus affects properties such as crystallinity, particles size and hardness etc. of as produced metal oxide. Alumina powder was undertaken as objective of study. Results analyzed are in agreement with literature data.

2. EXPERIMENTAL:

2.1 CHEMICAL COMPOSITION AND PARAMETERS:[6-8]

(a). ALUMINIUM NITRATE NON-HYDRATE(GR):

1	Symbol	Al $(NO_3)_3\ 9H_2O$
2	Oxidizing-reducing valency	-15
3	Solubility in distilled water	637g/liter at 25^0 C
4	Molecular Weight	375 g/mole

(b). UREA:

1	Symbol	$CO(NH_2)_2$
2	Oxidizing-reducing valence	+6
3	Solubility in distilled water	1080g/liter at 25^0 C
4	Molecular Weight	60 g/mole

(c) Glycine:

1	Symbol	$C_2H_5NO_2$
2	Oxidizing-reducing valency	+9
3	Solubility in distilled water	250g/litre at 25^0 C
4	Molecular Weight	75 g/mole

2.2 STOICHIOMETRIC OXIDIZER TO FUEL MOLAR RATIO[6-8]:

Al $(NO_3)_3$ (oxidizer)& $CO(NH_2)_2$ (fuel): Oxidizer/fuel ratio = 15/6 = 2.5 (From formula) i.e. : Stoichiometric aluminium nitrate: urea molar ratio is 1:2.5.

$Al(NO_3)_3$ (oxidizer) & $C_2H_5NO_2$ (fuel) : Oxidizer/fuel ratio = 15/9 = 1.66 (From formula) i.e. : Stoichiometric aluminium nitrate : Glycine molar ratio is 1:1.66.

2.3 PRECURSOR PREPARATION: Following are steps in preparation of precursor solution (Solution prepared after mixing of oxidizer & fuel before heat treatment):

1. Weighing of aluminium nitrate, urea and Glycine as per required quantity, calculated from formula.
2. Prepare solution with distilled water as per solubility of salts.
3. In last step homogeneous solution of Aluminium nitrate and fuel (to be used) was prepared separately. Three samples with variation from Stoichiometric value (as per formula of fuel to oxidizer ratio and ratio decided for a sample in proposed work.) were prepared and homogeneously mixed using magnetic stirrer.

Table 1: Samples detail (fuel to oxidizer ratio variation percentage).

Sample No.	Fuel used (Mixed Fuel)	Fuel level	Stoichiometric Fuel %
1M	(Urea+ Glycine)	Fuel lean	35+35=70 %
2M	(Urea+ Glycine)	Fuel lean	45+45=90 %
3M	(Urea+ Glycine)	Fuel Rich	60+60=120 %

The sample solutions were then subjected to heat treatment in electrical furnace. The value temperature was controlled in range of 300^{0}c - 450^{0}C, until formation of final product i.e. metal oxide.

2.4 SAMPLE PREPARATION FOR characterization: After combustion of solution in furnace (Heat treatment) foam like product formed in all of three samples. These samples were then grounded thoroughly to form a homogeneous powder. Powder samples were used for characterization (XRD, SEM).

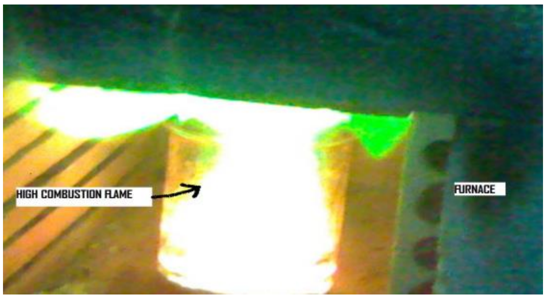
3. Results & discussion

3.1 Metal oxide formation and Combustion flame observation: Different colours of metal oxide product phase appeared after combustion process . Also flame content and duration has variation from fuel lean to fuel rich sample. White product formation is indication of complete combustion. Deviation of colour of product phase from white to black/brown may be due to residual carbon content from incomplete combustion reaction. High flame shows highly exothermic reaction and thus sufficient heat for formation of metal oxides.[5-8] Exothermicity of combustion process increases when we proceed from fuel lean to fuel rich sample.

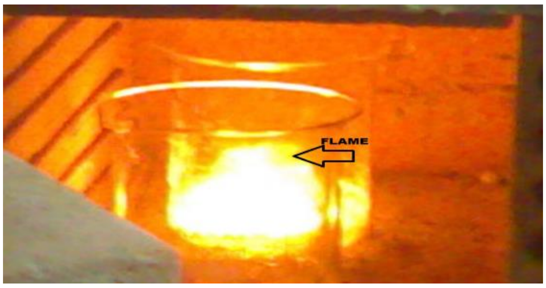
N. Sharma et. al. have reported similar behaviour of fuel to oxidizer ratio in production of metal oxide.[41]

Sample No.	Flame observation	
	Colour appearance	**Level/Duration**
1M	Brown-Black	Combustion progress with a little self originated flame that remains for about 3 seconds, only observed in some few area of sample.
2M	White (little brownish)	Combustion progress with a self originated flame that remains for about 13 seconds.
3M	White	Combustion progress with a self originated flame that remains for about 16 seconds. Flame raises high inside furnace. As product formed it starts rising inside container until combustion ends.

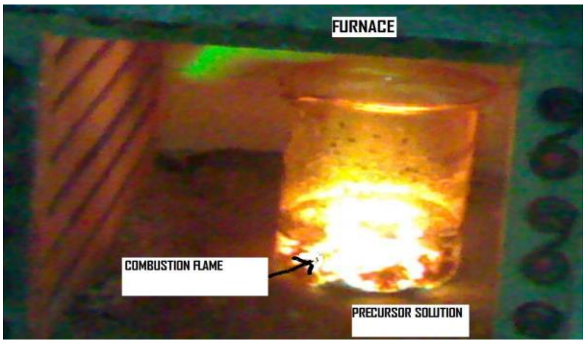
Table 2: observation of Combustion reaction in furnace.



(a)



(b)



(c)

Figure 1 : Combustion flame during formation of metal oxide (a) 1M (b) 2M (C) 3M

3.2 XRD ANALYSIS: From literature high flame indicates high exothermic combustion process. Large exothermicity of combustion process results in complete combustion of the reactant material. when the sufficient amount of energy required to achieve crystallization produces in combustion process, crystalline phase forms. This can be verified from XRD analysis of samples. In sample 1M as fuel lean configuration was used, amorphous product form because of lack of desired exothermicity during heat treatment. In case of samples 2M, 3M exothermicity of combustion increases with increase in fuel to oxidizer ratio.

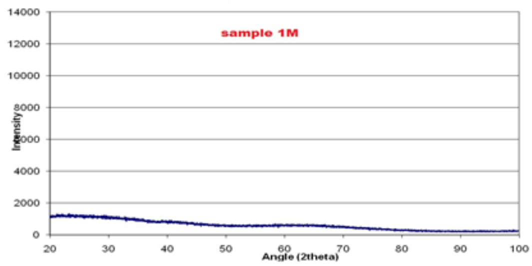


Figure 2: XRD of sample 1M (Fuel lean,70%)

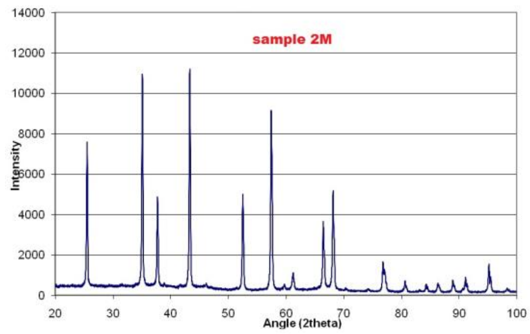


Figure 3: XRD of sample 2M (Fuel rich, 90%)

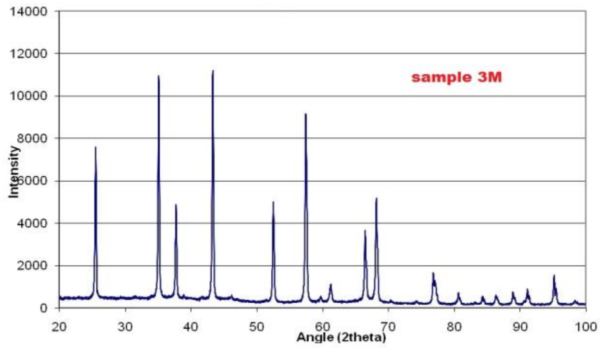


Figure 4 : XRD of sample 3M (Fuel rich, 70%)

3.3 MICROSTRUCTURE EXAMINATION: Range of particles size and formation of agglomerates is related to exothermicity of combustion process during production of metal oxide. If small range of particles size and a few agglomerates found in product phase, there will be insufficient heat produced during combustion. In case of sample 1M only few agglomerates with small range of metal oxide particles observed from microstructure study as shown below. In case of sample 2M, 3M foamy agglomerated particles with a wide distribution and presence of larger particles in their structure are found. All this is related to the amount of flame temperature during combustion process for production of metal oxide. Formation of large particles indicates high combustion flame. [41]

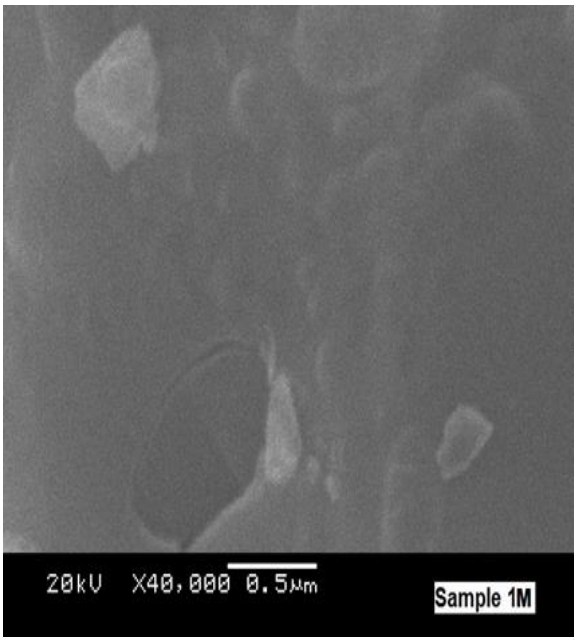


Figure 5: SEM micrograph of metal oxide samples (1M).

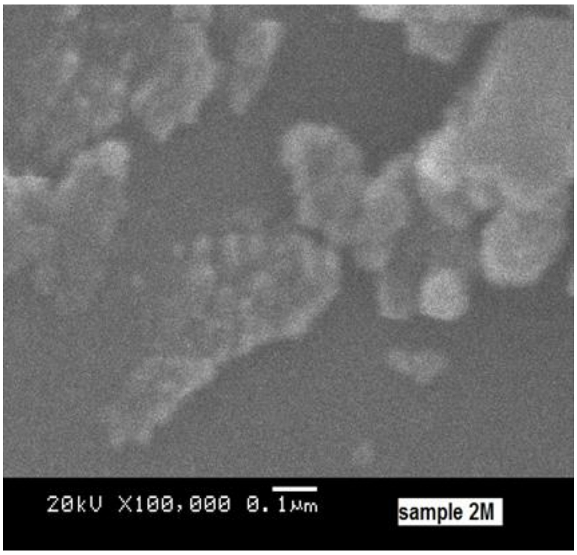


Figure 6 : SEM micrograph of metal oxide samples (2M).

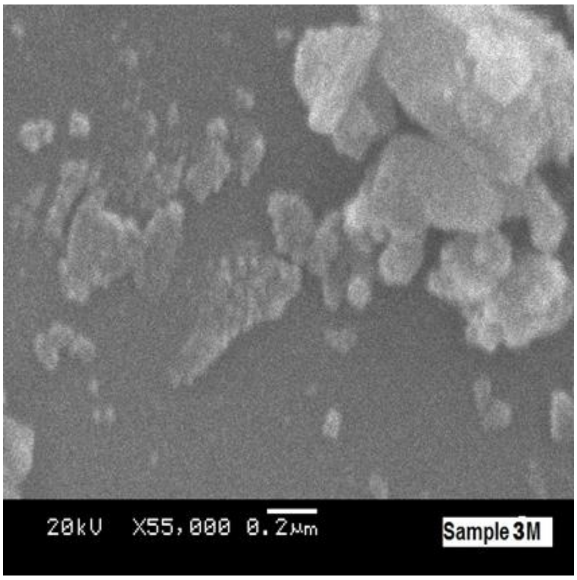


Figure 7: SEM micrograph of metal oxide samples (3M).

4. Conclusions

From production process of aluminium oxide (Alumina) and analysis of sample characterization we concluded that Fuel lean sample in lowest range of fuel to oxidizer ratio results in incomplete combustion, thus less exothermicity. Small range of exothermicity during heat treatment process gives rise to amorphous metal oxides, as in case of sample 1M. In case of little fuel lean and fuel rich sample trend of variation of exothermicity is as observed from literature. As the fuel to oxidizer ratio increases, crystallinity and exothermicity increases.Microstructure study confirms the extent of desired heat of combustion, by variation in range of particles size and formation of agglomerates.Overall from above analysis one can easily control parameters like mechanical properties (crystallinity or amorphous phase, hardness etc.), Particle size, and grade of metal oxide. Variation in fuel to oxidizer ratio leads to variation in properties of metal oxide formed (alumina powder in present study).

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