Synthesis and Characterization of Calcium Aluminate Spinel Using Solution Combustion Method

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Abstract— In this paper the Calcium aluminate nanoparticles were prepared by solution combustion method, using procured calcium nitrate, aluminium nitrate and urea (as fuel). Stoichiometric amounts of calcium nitrate, aluminium nitrate, and fuel urea were calculated using the total oxidizing and reducing valencies of the compounds which serve as numerical coefficients for stoichiometric balance. Initially Ca(NO3)2.4H2O and Al(NO3)3.9H2O were dissolved in a minimum quantity of water along with urea in a silica crucible (with a volume of 100cm³). The crucible was introduced into the muffle furnace which was preheated to 500°C. Also the variation of fuel to oxidizer (F/O) ratio was studied. The study was carried out for leaner to richer fuel ratios. In the case of CaAl2O4 combustion synthesis results were achieved when fuel (urea) was used. The use of fuel allowed the formation of pure, nano-crystalline CaAl2O4 directly from the combustion reaction. The effect of fuel ratios was investigated by variation of the ratios in which the fuel was taken.

Key words: Muffle furnace, solution combustion synthesis, CaAl2O4 Spinel, XRay-diffraction, SEM

I. INTRODUCTION

Calcium aluminate has been long involved in the practical applications in the cement and concrete industry, as well as in refractories and ceramic materials. Recently new advanced applications of calcium aluminate have appeared in optical and structural ceramics. Calcium aluminates (CA) are widely used in the steel and cement industry due to their relatively low density, hardness, straightness, etc. They are important constituents for refractory castables and govern their applicability to the service temperature. Calcium aluminate (CaAl2O4) has received considerable attention as a material for (implants) bone graft application due to its unique combination of physical and mechanical, bioactive and biocompatible properties. Also, CA-based glasses have technological importance in application of optical devices. Various synthesis methods have been developed to obtain CA with controlled particle size.

Traditionally calcium aluminate is produced by solid state reactions. The main drawback of this method is the required high temperature and the presence of multiple and not desired phases with the end product. Other methods are developed also using high temperatures. In order to synthesize mono-phase calcium aluminate, a long treatment period is required which leads to decreasing of surface area of product.

Park and Kim Referred to use boric acid as a flux to accelerate the formation of the product by solid state reactions of CaO and Al2O3 powders. The molten flux leads to uniform distribution of compounds and enhances diffusion of the precursors. This method enables to decrease the synthesis temperature. Many approaches are suggested to increase homogeneity of the precursors. Among them is sol–gel method. In this case precursors containing gel are prepared firstly. Later on, the gel is crystallized by subsequent heating at relatively low temperature. The most obvious advantage of the sol–gel method is that reagents are mostly mixed in atomic level, which may increase the reaction rate and decrease the synthesis temperature.

Douy and Gervais Referred a synthesis method of amorphous CaAl2O4 powders by spray-drying aqueous solutions of calcium and aluminum nitrates, followed by calcinations of product in order to completely decompose the salts. One of the well-known approaches of ceramic powder synthesis is Pechini method. The process is based on the ability of certain organic acids to chelate the metal ion. Heating the chelates, polymerification undergoes with the help of polyhydroxy alcohols and polymerized the resin forms. Then, the polymerized resin is calcined to obtain calcium aluminate. Also using chemical fixation of the cations by organic molecules Pati et al. and Gaki et al. have obtained nanoparticles of CaAl2O4 by versatile solution based precursor solution method.

The combustion method is a convenient method for the synthesis of oxide materials. It is a versatile, simple and rapid process, which allows to synthesize variety of materials without intermediate decomposition steps. This is a self-sustaining process and it is ignited in a temperature which is much lower than the actual phase transformation temperature. The calcium monoaluminate (CaAl2O4) has been synthesized with high purity via solution combustion methods, using aqueous solution of calcium and aluminium nitrates as both oxidizer and source of the corresponding cations and urea as a fuel. However, obtained products are mainly agglomerated, nevertheless this method do not need high temperatures as other methods.

The objective of this work is to study the influence of the quantity of urea on the possibility of synthesizing of calcium aluminate by the solution combustion synthesis method, using calcium and aluminium nitrates as precursors.

II. EXPERIMENTAL PROCEDURE

A. Synthesis of CaAl2O4 nanoparticle

The Calcium aluminate Nano powder were prepared by solution combustion method, using procured calcium nitrate, aluminium nitrate and urea (as fuel). Stoichiometric amounts of calcium nitrate, aluminium nitrate, and fuel urea were calculated using the total oxidizing and reducing valencies of the compounds which
serve as numerical coefficients for stoichiometric balance. Initially Ca(NO$_3$)$_2$.4H$_2$O and Al(NO$_3$)$_3$.9H$_2$O were dissolved in a minimum quantity of water along with urea in a crucible (with a volume of 100cm$^3$). The crucible was introduced into the muffle furnace which was preheated to 500°C.

![Muffle furnace](image1)

**Fig. 1: Muffle furnace**

![Crucible inside the muffle furnace](image2)

**Fig. 2: Crucible inside the muffle Furnace**

![Clear solution in crucible](image3)

**Fig. 3: Clear solution in crucible**

**B. Solution Combustion Synthesis (SCS) of Calcium Aluminate (CaAl$_2$O$_4$) with Fuel to Oxidizers Ratios:**

Calcium Nitrate, Aluminium Nitrate (Al(NO$_3$)$_3$.9H$_2$O) and urea (CO(NH$_2$)$_2$), were used as oxidizer and fuel respectively for solution combustion synthesis. These reactants are mixed in the required molar ratios with required amount in a minimum volume of deionized water to obtain clear aqueous solutions. The solution is kept in furnace preheated to 500°C. First thermal dehydration (at 100°C) takes place forming viscous solution, at 200°C the viscous liquids swelled and gets auto ignited, with the rapid evolution of a large volume of gases to produce voluminous powders. The nature of ignition depended on the fuel-to-oxidant ratio. The auto ignition of the precursor containing fuel-to-oxidant ratio according to the concept of propellant chemistry and fuel rich ratio was found to be more violent compared to the fuel-deficient precursor. Because the time for which the auto ignition exists is rather small (typically 5 s), under the equilibrium conditions the standard reaction equation in this systems can be represented for all trivalent nitrates - urea combustion (Where M=trivalent cation) for fuel to oxidizer ratio as For all Calcium nitrate, Aluminium nitrate, urea, combustion for F/O ratios equation can be written as.

\[
\text{Ca(NO}_3\text{)}_2\cdot4\text{H}_2\text{O} + 2\text{Al(NO}_3\text{)}_3\cdot9\text{H}_2\text{O} + 6.66\phi\text{NH}_2\text{CONH}_2 \rightarrow \text{CaAl}_2\text{O}_4 + (4+6.66\phi)\text{N}_2 + 6.66\phi\text{CO}_2 + (22+13.32\phi)\text{H}_2\text{O} + (10-10\phi)\text{O}_2 \quad (1)
\]

Here the $\phi$ is the fuel to oxidizer equivalence ratio (not the urea to nitrate molar ratio) given by the formula

\[
\text{Fuel Ratio} = \frac{\sum \text{all oxidising and reducing elements in fuel}}{\sum \text{all oxidising and reducing elements in oxidizer}}
\]

![Different powders formed after SCS with fuel to oxidizer (F/O) variation when urea was used as fuel.](image4)

**Fig. 4: Different powders formed after SCS with fuel to oxidizer (F/O) variation when urea was used as fuel.**

![a) No flame is formed when the F/O=0.4 inside the muffle furnace](image5)

**Fig. 5: a) No flame is formed when the F/O=0.4 inside the muffle furnace**

![b) No flame is formed when the F/O=0.8 inside the muffle furnace](image6)

**Fig. 5.b) No flame is formed when the F/O=0.8 inside the muffle furnace**
C. Structural, Compositional and Micro-structural Analysis.

1) X-ray diffraction (XRD)
X-ray diffraction studies were carried out for phase confirmation and calculating crystallite size of the milled samples, using D8-Advance-Bruker machine with Cu-Kα (wavelength of Cu-Kα (λ) \( \sim1.5406 \) Å) radiations for all the measurements. Ni filter was used to attenuate Kβ lines. The crystallite size of powders was calculated using Scherrer’s formula where an assumption was made that the particle is spherical in shape. The Scherrer’s formula gives

\[
\beta_0 = \frac{K' \lambda}{L \cos \theta}
\]

where, \( \beta_0 \) is the full width at half maximum (FWHM) of diffracted peaks in degrees, \( L \) stands for the liner dimension of particles in meters, \( \theta \) refers to Bragg’s angle in degrees and \( K' \) is the shape factor, generally known as a numerical constant and evaluated as 0.93 and depends on shape of crystallites. From this expression, it is clearly seen that diffracted beam gets broadened as the size of crystallite reduces. Silicon was used as an external standard for correction due to instrumental broadening. The XRD Machine is shown in Figure 6.

2) Scanning electron microscope (SEM)
The surface morphology of powders and pellet samples was studied using scanning electron microscope a Cambridge, stereoscan-240. (ESEM –FEI, Quanta 200) and elemental compositional analysis was done using the attached energy dispersed analysis of X-rays (EDAX). The grain size of the chosen materials was seen clearly using FEG-SEM since it produces the primarily high electron brightness at low accelerating potentials. All the samples studied were ceramic oxide insulator materials (non conductors), the gold coating was done to avoid the charge accumulation on sample surface by making path way to the ground. Grain size was estimated using linear intercept method. The scanning Electron Microscope(SEM) is as shown in figure 7.

D. Thermodynamic Modeling
For comparing exothermicity of fuel in reaction with the nitrates, enthalpy and adiabatic flame temperature of the
reactions between urea and aluminum nitrate were calculated in the conditions Stoichiometric, Fuel lean and Fuel rich. Using the thermodynamic data for the various reactants and products listed in Table 1, the enthalpy of combustion and the theoretical adiabatic flame temperatures as a function of F/O ratio were approximately calculated by the following equations: using Mathematical software.

\[
\Delta H^0 = \left(\sum_n \Delta H_f^0\right)_{\text{products}} - \left(\sum_n \Delta H_f^0\right)_{\text{reactants}} \tag{3}
\]

<table>
<thead>
<tr>
<th>compound</th>
<th>(\Delta H_f^0) (kcal mol(^{-1}))</th>
<th>(c_p) (cal mol(^{-1}) K(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaAl(_2)O(_4)</td>
<td>-534</td>
<td>42.79</td>
</tr>
<tr>
<td>Al((\text{NO}_3))(_3) (c)</td>
<td>-857.59</td>
<td>-</td>
</tr>
<tr>
<td>Zn((\text{NO}_3))(_2):6(\text{H}_2)O (c)</td>
<td>-551.30</td>
<td>-</td>
</tr>
<tr>
<td>NH(_2)CONH(_2) (c)</td>
<td>-79.71</td>
<td>-</td>
</tr>
<tr>
<td>NH(_2)CH(_2)COOH (c)</td>
<td>-126.22</td>
<td>-</td>
</tr>
<tr>
<td>CH(_2)(CH(_2))(_2)COH OH (c)</td>
<td>-100.26</td>
<td>-</td>
</tr>
<tr>
<td>Ca(NO(_3))(_2):4(\text{H}_2)O (c)</td>
<td>-509.64</td>
<td>-</td>
</tr>
<tr>
<td>Mg((\text{NO}_3))(_2):6(\text{H}_2)O (c)</td>
<td>-217.75</td>
<td>-</td>
</tr>
<tr>
<td>SiO(_2)</td>
<td>-70.52</td>
<td>-</td>
</tr>
<tr>
<td>Ca(_2)(_3)(_2)(_2)(_2)O(_9) (ODH)</td>
<td>-28.8</td>
<td>-</td>
</tr>
<tr>
<td>C(_6)H(_2)N(_2) (HMT)</td>
<td>-764.447</td>
<td>52.3+0.00774T</td>
</tr>
<tr>
<td>CaMg(_2)Si(_2)O(_6) (c)</td>
<td>-399.09</td>
<td>22.08+0.0089T</td>
</tr>
<tr>
<td>NH(_2)NO(_3)</td>
<td>-83.24</td>
<td>11.40+0.00145T - 182400/T(^2)</td>
</tr>
<tr>
<td>CO(_2) (g)</td>
<td>-94.051</td>
<td>10.34+0.00274T - 195500/T(^2)</td>
</tr>
<tr>
<td>NO(_2) (g)</td>
<td>7.93</td>
<td>8.8</td>
</tr>
<tr>
<td>N(_2) (g)</td>
<td>0</td>
<td>6.50+0.0010T</td>
</tr>
<tr>
<td>H(_2)O (g)</td>
<td>-57.796</td>
<td>8.22+0.00015T + 0.00000134T(^2)</td>
</tr>
<tr>
<td>H(_2)O (l)</td>
<td>-68.38</td>
<td>-</td>
</tr>
<tr>
<td>O(_2) (g)</td>
<td>0</td>
<td>8.27+0.00258T - 18770/T(^3)</td>
</tr>
<tr>
<td>C (c)</td>
<td>0</td>
<td>2.673+0.002617T - 116900/T(^3)</td>
</tr>
<tr>
<td>C((\text{carbon}))</td>
<td>0</td>
<td>2.038</td>
</tr>
<tr>
<td>HCNO(g)</td>
<td>40.88</td>
<td>11.21</td>
</tr>
<tr>
<td>NH(_2) (g)</td>
<td>-10.96</td>
<td>-</td>
</tr>
<tr>
<td>N(_2)O(_5) (g)</td>
<td>19.5</td>
<td>-</td>
</tr>
</tbody>
</table>

| (c):crystalline; (g):gas; (l):liquid; (T):absolute temperature; |

Table 1: Relevant thermodynamics data of reactants and products

III. RESULTS AND DISCUSSION

Solution Combustion Synthesis (SCS) of Calcium Aluminate (CaAl\(_2\)O\(_4\)) with Various F/O Ratios:

Considering the reaction of nitrates with the urea fuel the obtained results are, For fuel lean Ø=1, three compositions of fuel lean are Ø=0.4, 0.8 and chemical equations are respectively (60%, 20% fuel lean) Ø=0.8, fuel lean.

Ca(NO\(_3\))\(_2\):4\(\text{H}_2\)O +2\(\text{Al}\) (NO\(_3\))\(_3\):9\(\text{H}_2\)O+2.66NH\(_2\)CONH\(_2\)

\[\rightarrow\text{CaAl}_2\text{O}_4+6.66\text{N}_2+2.66\text{CO}_2+27.32\text{H}_2\text{O}\]

Ø=0.8 fuel lean

Ca(NO\(_3\))\(_2\):2\(\text{H}_2\)O +2\(\text{Al}\) (NO\(_3\))\(_3\):9\(\text{H}_2\)O+5.33NH\(_2\)CONH\(_2\)

\[\rightarrow\text{CaAl}_2\text{O}_4+9.33\text{N}_2+5.33\text{CO}_2+32.66\text{H}_2\text{O}\]

Fuel stoichiometric ratio Ø=1

Ca(NO\(_3\))\(_2\):4\(\text{H}_2\)O +2\(\text{Al}\) (NO\(_3\))\(_3\):9\(\text{H}_2\)O+6.66NH\(_2\)CONH\(_2\)

\[\rightarrow\text{CaAl}_2\text{O}_4+10.66\text{N}_2+6.66\text{CO}_2+35.32\text{H}_2\text{O}\]

Fuel rich Ø>1 Ø=1.2, 1.4, 1.6 (20%, 40%, 60% fuel rich) reaction equations are

Ca(NO\(_3\))\(_2\):4\(\text{H}_2\)O +2\(\text{Al}\) (NO\(_3\))\(_3\):9\(\text{H}_2\)O+8NH\(_2\)CONH\(_2\)

\[\rightarrow\text{CaAl}_2\text{O}_4+12\text{N}_2+8\text{CO}_2+53\text{H}_2\text{O}\]

Ca(NO\(_3\))\(_2\):4\(\text{H}_2\)O +2\(\text{Al}\) (NO\(_3\))\(_3\):9\(\text{H}_2\)O+9.33NH\(_2\)CONH\(_2\)

\[\rightarrow\text{CaAl}_2\text{O}_4+13.33\text{N}_2+9.33\text{CO}_2+42.66\text{H}_2\text{O}\]

Ca(NO\(_3\))\(_2\):4\(\text{H}_2\)O +2\(\text{Al}\) (NO\(_3\))\(_3\):9\(\text{H}_2\)O+10.66NH\(_2\)CONH\(_2\)

\[\rightarrow\text{CaAl}_2\text{O}_4+14.66\text{N}_2+10.66\text{CO}_2+45.32\text{H}_2\text{O}\]

Table 2: Amount of fuel and oxidizer. Physical characteristics of the combustion reaction for different Urea-to-nitrate ratios (Fuel- oxidizer ratios)

A. Adiabatic Flame Temperature and Enthalpy Calculations for Urea as Fuel

1) Ø=0.4, fuel lean.

\[\Delta H_{\text{Products}} = \Delta H_{\text{(CaAl}2\text{O}4)} + 6.66 \cdot \Delta H_{\text{N}_2} + 2.66 \cdot \Delta H_{\text{CO}_2} + 27.32 \cdot \Delta H_{\text{H}_2\text{O}} \]

\[\Delta H_{\text{Products}} = -534 + 6.66 \cdot 94.051 + 2.66 \cdot 857.59 + (-57.796) = -2363.15 \text{ kcal mol}^{-1} \]

2) Ø=0.8, fuel lean.

\[\Delta H_{\text{Products}} = -534 + 6.66 \cdot 94.051 + 2.66 \cdot 857.59 + (-57.796) = -2363.15 \text{ kcal mol}^{-1} \]

\[\Delta H_{\text{Products}} = -509.64 + 2 \cdot (-857.59) + 2.66 \cdot (-79.71) = -2436.20 \text{ kcal mol}^{-1} \]

\[\Delta H_{\text{Products}} = -2363.15 - (-2436.20) = 73.05 \text{ kcal mol}^{-1} \]

\[C_p = 42.79 + 6.66 \cdot 9.61 + 2.66 \cdot 8.87 + 27.32 \cdot 8.025 = 331.22 \text{ kcal mol}^{-1} \]

\[\Delta H_{\text{Reactions}} = \Delta H_{\text{Products}} - \Delta H_{\text{Reacting}} \]

\[\Delta H_{\text{Products}} = 298 + (73.05 \cdot 331.22) \cdot 1000 = 518.54 \]

\[\Delta H_{\text{Reactions}} = -2922.9 \text{ kcal mol}^{-1} \]

\[\Delta H_{\text{Reactions}} = -2922.9 \text{ kcal mol}^{-1} \]

\[\Delta H_{\text{Reactions}} = -2922.9 \text{ kcal mol}^{-1} \]
\[ C_p = 42.79 + 9.33x6.961 + 5.33x8.87 + 32.66x8.025 = 418.40 \text{cal mol}^{-1} \]

\[ T_{ad} = T_o + (\Delta H_p - \Delta H_r) / C_p = 298 + (273/418.4) \times 1000 = 950.48 \]

3) Fuel stoichiometric ratio \( \theta = 1 \)

Ca(NO\(_3\))\(_2\)+2Al(NO\(_3\))\(_3\)+9H\(_2\)O+6.66NH\(_2\)CONH\(_2\)

\[ \rightarrow \text{CaAlO}_2+10.6N_2+6.66\text{CO}_2+35.32\text{H}_2\text{O} \]

\[ \Delta H (\text{Products}) = \Delta H (\text{CaAl}_2\text{O}_3)+15.6^\circ \Delta H _{N_2}+6.6^\circ \]

\[ \Delta H (\text{CO}_2)+35.32^\circ \Delta H _{H_2O} \]

\[ \Delta H (\text{Products}) = -534+10.6^\circ 0+6.6^\circ (-94.051)+35.32^\circ = -3201.72 \text{ kcal mol}^{-1} \]

\[ \Delta H (\text{Reactants}) = \Delta H (\text{Ca(NO}_3)_2\text{2H}_2\text{O}) + 2^* \Delta H (\text{Al(NO}_3)_3\text{9H}_2\text{O}) + 6^* \Delta H (\text{NH}_2\text{CONH}_2) \]

\[ \Delta H (\text{Reactants}) = -509.64 + 2^*(-857.59) + 6.6^*(-79.71) = -2755.66 \text{ kcal mol}^{-1} \]

\[ \Delta H (\text{Reactants}) = \Delta H (\text{Products}) - \Delta H (\text{Reactants}) \]

\[ \Delta H (\text{Reactants}) = -3201.72 - (-2755.66) = -446.06 \text{ kcal mol}^{-1} \]

\[ C_p = 42.79 + 10.66\text{6.66x8.87}+35.32x8.025 = 433.39 \text{ cal mol}^{-1} \]

\[ T_{ad} = T_o + (\Delta H_p - \Delta H_r) / C_p = 298 + (965.20/549.97) \times 1000 = 1944.33 \]

<table>
<thead>
<tr>
<th>S No</th>
<th>F/O ratio</th>
<th>Urea/nitrate molar ratio</th>
<th>Enthalpy Kcal/mol</th>
<th>Adiabatic flame Temp C</th>
<th>No gases evolved</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.4</td>
<td>0.5</td>
<td>73.05</td>
<td>518.54</td>
<td>46.5</td>
</tr>
<tr>
<td>2</td>
<td>0.8</td>
<td>1.0</td>
<td>-273</td>
<td>9504.8</td>
<td>57.5</td>
</tr>
<tr>
<td>3</td>
<td>1.0</td>
<td>1.5</td>
<td>-446.06</td>
<td>1327.23</td>
<td>62.5</td>
</tr>
<tr>
<td>4</td>
<td>1.2</td>
<td>2</td>
<td>-619.74</td>
<td>1533.73</td>
<td>67.5</td>
</tr>
<tr>
<td>5</td>
<td>1.4</td>
<td>2.5</td>
<td>-792.96</td>
<td>1741.97</td>
<td>75</td>
</tr>
<tr>
<td>6</td>
<td>1.6</td>
<td>3</td>
<td>-965.20</td>
<td>1944.33</td>
<td>79.5</td>
</tr>
</tbody>
</table>

Table: 3 Effect of Urea – Nitrate F/O ratio on adiabatic flame temperature, enthalpy of reaction, no of moles of gases evolved

\[ \Delta H (\text{Reactants}) = -509.64 + 2^*(-857.59) + 10.6^*(-79.71) = -3074.52 \text{ kcal mol}^{-1} \]

\[ \Delta H (\text{Reactants}) = \Delta H (\text{Products}) - \Delta H (\text{Reactants}) \]

\[ \Delta H (\text{Reactants}) = -4039.72 - (-3074.52) = -965.20 \text{ kcal mol}^{-1} \]

\[ C_p = 42.79 + 14.66\text{6.66x8.87}+43.32x8.025 = 586.28 \text{ cal mol}^{-1} \]

\[ T_{ad} = T_o + (\Delta H_p - \Delta H_r) / C_p = 298 + (965.20/586.28) \times 1000 = 1444.33 \]

Fig. 8: Graph representing the enthalpy vs F/O ratio

For urea as a fuel

\[ \Delta H (\text{Reactants}) = \Delta H (\text{Products}) - \Delta H (\text{Reactants}) \]

\[ \Delta H (\text{Reactants}) = -4039.72 - (-3074.52) = -965.20 \text{ kcal mol}^{-1} \]

\[ T_{ad} = T_o + (\Delta H_p - \Delta H_r) / C_p = 298 + (965.20/586.28) \times 1000 = 1444.33 \]

Fig. 9: Graph representing the Adiabatic temperature v/s F/O ratio For urea as a fuel
articles are sintered to form big
ue
n of Calcium
rnal of Chemical
ith urea as
ures (500 ºC)
nd the
Figure 11

Aggregates of 1
reaction. Some par
escaped number of moles
from 10 to100 nm.
porous morphology with
Figure 11

Break and form the porous
structure as seen

The agglomerates are porous d
O
O
m. Some isolated 100nm seized
2
2
2
2

O
2
4
O
2
4

Ca
Ca
Al
Al

Ca

Al

Some particles are sintered to form big hard
agglomerates of 1-5µm. Some isolated 100nm seized particles
\textit{are also seen in CaAl}_2O_4 powder. The formation of CaAl_2O_4
when used with urea as fuel shows that temperature given
was enough to help the formation of the spinel powder.
Associated gas evolution results in highly porous structure
as the amount of gas increases agglomerates are more likely
to break and form the porous structure as seen in the images.

B. Scanning Electron Microscope Observations

Figure 11 and 12 show the SEM images of CaAl_2O_4 of
porous morphology with plate like agglomerates ranging
from 10 to100 nm. The agglomerates are porous due to
escaped number of moles of gases released in combustion
reaction. Some particles are sintered to form big hard
agglomerates of 1-5µm. Some isolated 100nm seized particles
are also seen in CaAl_2O_4 powder. The formation of CaAl_2O_4
when used with urea as fuel shows that temperature given
was enough to help the formation of the spinel powder.
Associated gas evolution results in highly porous structure
as the amount of gas increases agglomerates are more likely
to break and form the porous structure as seen in the images.

IV. CONCLUSIONS

From the undertaken studies it was concluded that the
amount of urea has a big influence on the morphological
characteristic and phase composition of the product obtained
by solution combustion synthesis. Although, the system
comes to the equilibrium and pure calcium aluminate forms
using stoichiometric amount of urea.

The end results are promising and the characterization results prove that the formation of Calcium Aluminate can be carried out at low temperatures (500 ºC)
using urea as fuel. More precisely it is a low cost and newer
method of producing oxides.

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