Nano-catalyst Synthesis by Flame Spray Pyrolysis

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Abstract—Nano size perovskite type catalysts were prepared by Flame Spray Pyrolysis (FSP) method. A commercial nozzle was inserted inside a custom made premixed burner, surrounded by 22 methane flamelets. Liquid precursors were sprayed into the center of the flamelets ring to produce the nano particles. Collection of the particles was achieved on GFA filters by applying vacuum. Particles were characterized by Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) techniques for their morphology and size. Phase compositions were determined by X-Ray Diffraction (XRD) method.

Keywords— flame spray pyrolysis, nano particles, perovskites, catalysis

I. INTRODUCTION

The incentive to produce catalyst particles in nano size is to maximize the surface area of the active phase for reactions, providing a higher reaction rate as well as a significant cost reduction. The most important commercial products produced by combustion synthesis are carbon black (Cabot, Degussa, Columbia), fumed silica (Cabot, Degussa), pigmentary titania (DuPont, Millenium, Kerr-McGee) and optical fibers (Corning, Lucent, Sumitomo) [1].

Material production by flame synthesis is studied by two methods in laboratory scale; flame aerosol synthesis (or flame hydrolysis) and flame spray pyrolysis (FSP). In flame aerosol method, metal precursors are dissolved in an appropriate solvent and the solvent vapor is carried into the flame environment by a carrier gas followed by a gas to particle conversion. Flame spray pyrolysis on the other hand is more versatile as it uses liquid precursors producing particles of more complex compositions. Controlled synthesis of particles is very important in FSP as small variations in process conditions change the particle quality.

II. EXPERIMENTAL PROCEDURE

The objective of this work is to prepare nano size perovskite type catalysts by FSP method. The schematic of the experimental system is shown in Figure 1. Metal precursors are dissolved in an appropriate solvent and carried into the flame environment by a syringe pump and a capillary nozzle, followed by a particle collection system.

Methane and oxygen flowrates of 2 and 4.5 LPM provided stable flamelets as shown in Figure 2a. Metal precursors dissolved in propionic acid were injected into the flame and dispersed by oxygen dispersion gas (Figure 2b). The effects of liquid precursor feed rate, dispersion gas flow rate and the metal/solvent ratio on the flame stability, particle quality and quantity were investigated. Active catalyst compositions that had been determined for CH₄ combustion previously (prepared by sol-gel citrate method) [2] were prepared by the FSP method in this study.
III. RESULTS AND DISCUSSION

X-Ray Diffraction analysis and Scanning Electron Microscopy images of LaCoO$_3$, produced under the conditions given in Table 1, are shown in Figure 3 and 4.

Table 1: Test conditions at solution flow rate of 4 RPM

<table>
<thead>
<tr>
<th>Test No</th>
<th>Metal/Solvent Ratio (mol/mol)</th>
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</thead>
<tbody>
<tr>
<td>T1</td>
<td>0.100</td>
</tr>
<tr>
<td>T2</td>
<td>0.074</td>
</tr>
<tr>
<td>T3</td>
<td>0.500</td>
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</table>

XRD analysis shows that the main phase obtained is perovskite, with a minor amount of Co$_3$O$_4$.

Figure 3: XRD spectrum of the LaCoO$_3$ catalyst

- (●) LaCoO$_3$
- (■) Co$_3$O$_4$

SEM pictures of the catalysts as prepared are presented in Figure 4. They all show spherical-like particles with varying degrees of agglomeration. At higher magnitudes, it was seen that, all the particles were under 100 nm. TEM pictures in Figure 5 showed that, the primary particle sizes were under 20 nm. At lower metal to solvent ratio (mol/mol), particles as small as 5 nm were observed. In SEM and TEM pictures, egg-shell structures were observed. These structures indicate the existence of droplet-to particle formation mechanism, along with the nucleation from the gas phase. The EDX analysis of
TEM pictures showed clear crystal planes, indicating the presence of crystalline domains.

Figure 4: SEM pictures of the LaCoO₃ catalyst

Figure 5: TEM pictures of the LaCoO₃ catalyst
ACKNOWLEDGMENT

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REFERENCES