

# Synthesis of nanometre Sized Pd-CoO a Transition Bimetal Oxide using Sonochemical Method and its Characterization

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**Abstract:** Sonochemical method has been applied for the synthesis of transition metal oxide nanoparticle from their metal salts. The Pd-CoO synthesized is characterized using X-ray diffraction (XRD), Energy Dispersive X-ray Spectroscopy (EDX), Scanning Electron microscopy (SEM) and Atomic Force microscopy (AFM). By this all different technique for characterization we got its particle size and its morphology. Method applied and procedure followed for the synthesis has been discussed in detail.

**Keywords:** Sonochemical method, acoustic cavitation, Nanometre size, transition Bimetal oxide, Characterization

## 1. Introduction

[1] Metal salts are very useful reagents and play an important role in the synthesis of some industrially important metal oxides catalyst. Size of the particle always plays an important role in the determining its chemical and physical properties. So nanometre size of the particle has shown great effect. Oxides of the transition metal are very oxidation catalyst and when they are present in Nano size they became highly reactive. Due to the larger surface volume ratio they shows a characteristic properties and have many applications such as gas sensors, magnetic storage media, solar energy transformation, semiconductors and organic catalysis. The use of alternate energy like ultrasound energy could be explored for this purpose as the use of ultrasound is expected to shorten the reaction time and minimize the requirement of chemicals used for the synthesis of materials. This is due to a phenomenon called *acoustic cavitation*, which generates very high temperature, pressure and turbulence in liquid being irradiated by ultrasound.

## 2. Experimental Section

1gm of cobalt sulphate is dissolved in 80ml dist. Water in one beaker. And then then in another beaker 0.1gm of palladium acetate is dissolved in 20ml ethanol. Then after dissolving both the sample properly, both the solution is mixed together. And 1gm of PVP is added to the mixed solution and Sonicated under direct sonication for 30mins. Initial pH of solution = 3.30 after 30mins black solution is obtained which dried at 110degree to get powder.

Sonication Parameter

1. Pulse : 05 ON / 02 OFF
2. Amp : 30%
3. Time : 30mins
4. Set Point Temperature : 70degree
5. Probe Temperature : 25degree

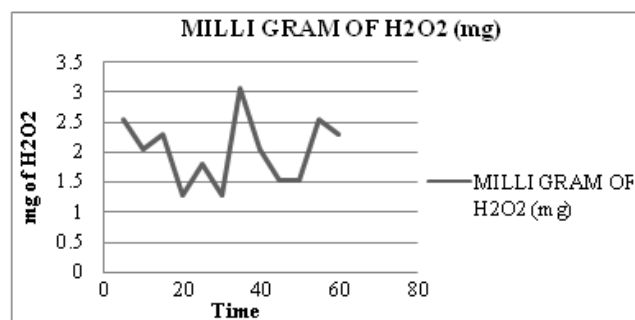
## 3. H<sub>2</sub>O<sub>2</sub> Estimation

Process Conditions

1. Catalyst used is pd-CoO powder.
2. Under Sonication condition of Pulse : 05 ON / 02 OFF, Amp : 30%, Time : 30mins, Set Point Temperature : 70degree, Probe Temperature : 25degree

**Table 1:** For H<sub>2</sub>O<sub>2</sub> Estimation

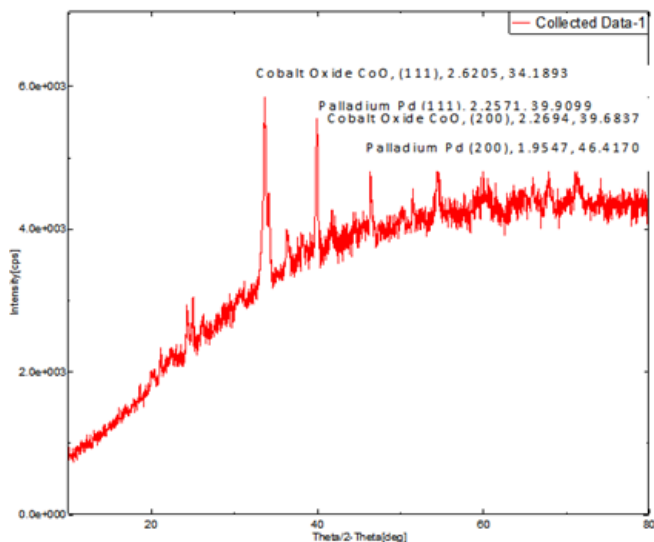
Time	KMnO <sub>4</sub> Titre	Mole of H <sub>2</sub> O <sub>2</sub> (μmole)	Milli Gram of H <sub>2</sub> O <sub>2</sub> (mg)
5	1.0	75	2.55
10	0.8	60	2.04
15	0.9	67.5	2.295
20	0.5	37.5	1.275
25	0.7	62.5	1.785
30	0.5	37.5	1.275
35	1.2	90	3.06
40	0.8	60	2.04
45	0.6	45	1.53
50	0.6	45	1.53
55	1.0	75	2.55
60	0.9	67.5	2.295



**Figure 1:** Plot of mg H<sub>2</sub>O<sub>2</sub> Vs. Time

## 4. Results

4.1 XRD plot of the catalyst Pd-CoO



Sample	Peaks (Selected)	2 Theta (deg)	d(A°)	FWHM	Intensity
Palladium	1	39.9099	2.25725	0.2233	424.38
	2	46.4176	1.95434	0.2642	211.46
Cobalt oxide	1	34.1893	2.62049	0.399	499.15
	2	39.6837	2.25725	0.2233	681.48

Figure 3: XRD image shows the presence of palladium and cobalt oxide at different peaks

Table 2: For XRD peaks of Pd-CoO

4.2 SEM Figure of Pd-CoO

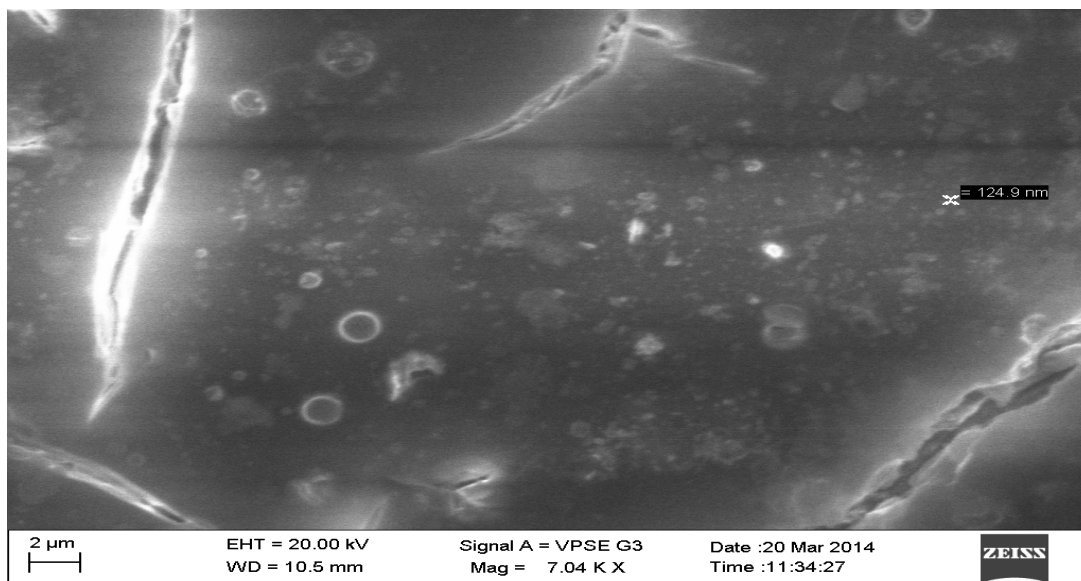


Figure 3: SEM image shows size of particle 124.9nm at 2μm

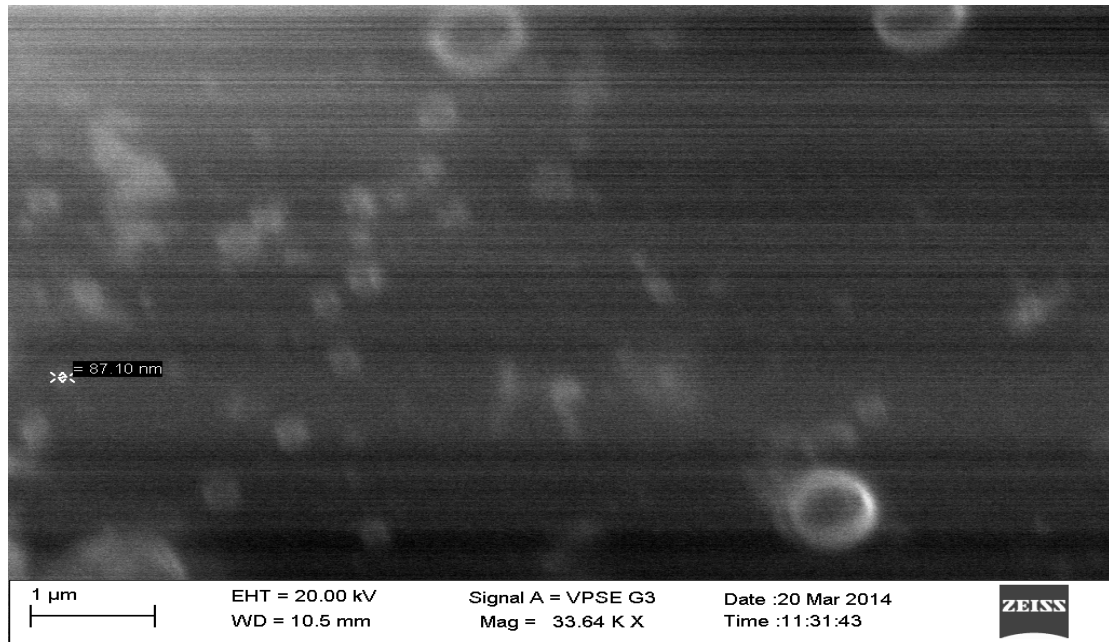


Figure 4: SEM image shows size of particle 87.10nm at 1µm.

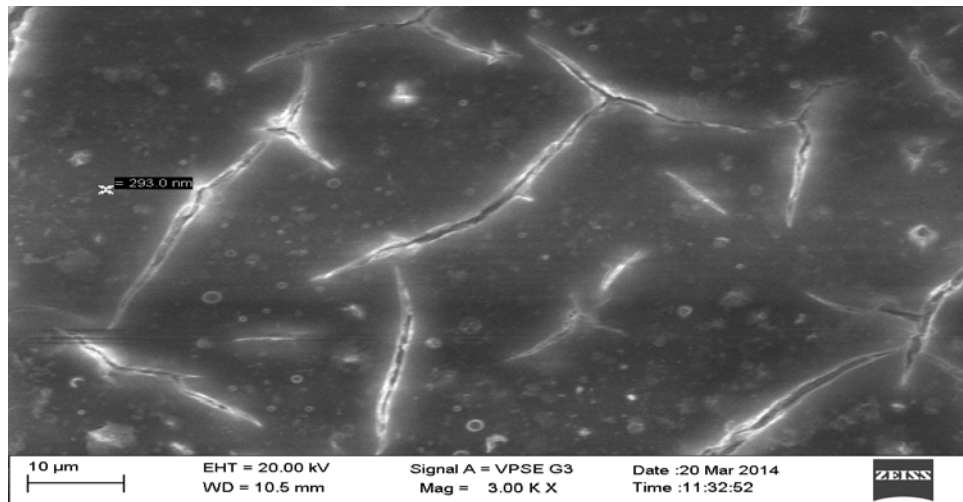
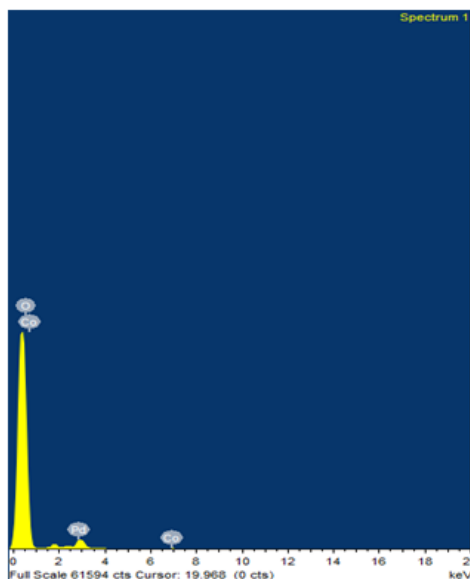
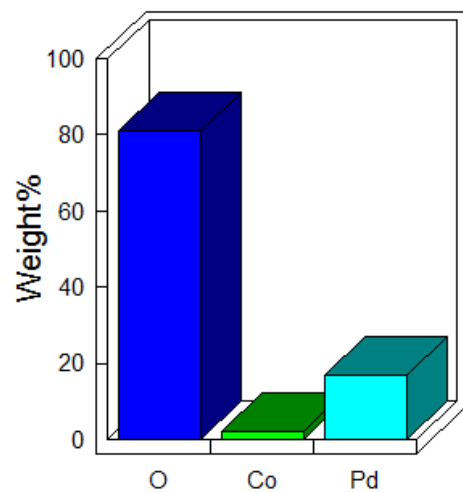


Figure 5: SEM image shows size of particle 293nm at 10µm.

#### 4.3 EDX Report of Pd-CoO



#### Quantitative results



Spectrum processing: No peaks omitted  
 Processing option: All elements analyzed (Normalised)  
 Number of iterations = 8

Standard:

O SiO2 1-Jun-1999 12:00 AM

Co Co 1-Jun-1999 12:00 AM

Pd Pd 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
O K	80.92	96.29
Co K	2.07	0.67
Pd L	17.01	3.04
Totals	100.00	

4.4 AFM Report of Pd-CoO

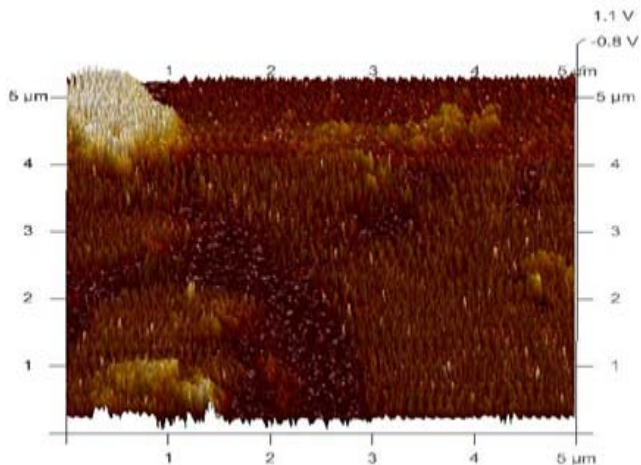


Figure 7: AFM image Pd-CoO

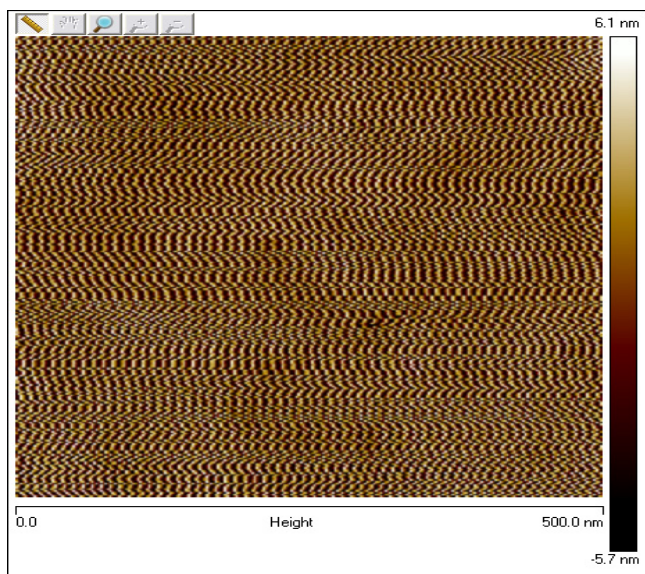


Figure 8: 2-D Image of AFM of Pd-CoO

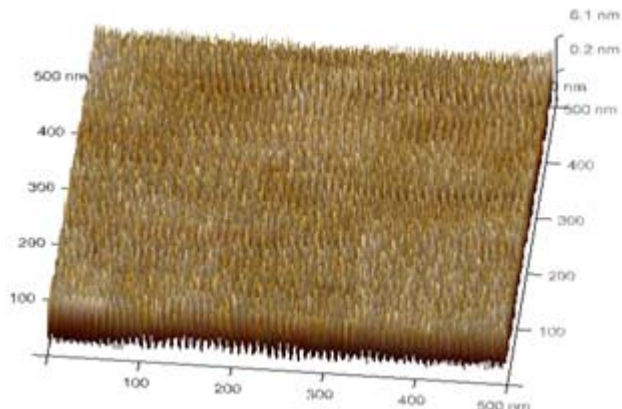


Figure 9.3: D Image of AFM of Pd-CoO AFM Report

Table 3: Shows AFM Reports of Pd-CoO

Parameter	Mean	Minimum	Maximum	Sigma
Total Count	10.000	10.000	10.000	0.000
Density	2.500(/ $\mu\text{m}^2$ )	2.500(/ $\mu\text{m}^2$ )	2.500(/ $\mu\text{m}^2$ )	0.000(/ $\mu\text{m}^2$ )
Height	137.210 (nm)	34.739 (nm)	213.112(nm)	44.166 (nm)
Area	2038.574 (nm <sup>2</sup> )	1525.879 (nm <sup>2</sup> )	2807.617 (nm <sup>2</sup> )	354.004 (nm <sup>2</sup> )
Diameter	50.763 (nm)	44.777 (nm)	59.789 (nm)	4.322 (nm)

5. Discussion and Conclusion

[3] Mechanism involved in the formation of Pd-CoO is intense ultrasonic wave produced during the sonication which is strong enough to produce the cavitation which increases the rate of oxidation, reduction, dissolution and polymerization reactions. These all reactions are due to the collapse of bubble in aqueous media. High pressure and high temperature are produced which causes the pyrolysis of water into H and OH radicals. And there are many more reasons which increase the rate of reaction and help in formation of Nano size particle. <sup>5</sup>During the characterization of the catalyst we found the SEM report that the size of the catalyst is 87.10nm. And also under AFM we have found the average dia and mean dia of the particle is nanometre size. Hence we can say that from all the above results which we have got that particle size can be reduced to Nano size and this Nano catalyst can be used for applications.

References

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