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Synthesis of CuO nanoparticles using copper nitrate hydrate, urea and sucrose by solution combustion synthesis

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Abstract

CuO nanoparticles were prepared by using solution combustion synthesis with copper nitrate as an oxidizer, urea as the fuel, and sucrose as the additive fuel. Optimum fuel-to-oxidizer ratio(Φ_e) and temperature for the synthesis of CuO nanoparticle was determined. The synthesized CuO oxides were characterized by X-ray diffraction (XRD) spectroscopy, scanning electron microscopy (SEM), scanning probe microscopy(SPM) and Brunauer–Emmet–Teller (BET) techniques. The phase, grain size, morphology, particle size and specific surface area of CuO oxide nanoparticles were found to be dependent on the fuel to oxidizer ratio(Φ_e) and temperature. The optimum synthesis conditions are Φ_e=0.2 and T=400°C, and the properties of the CuO nanoparticles obtained under this condition are 104nm in average particle size, 70.15 m^2/g in BET specific surface area, and present in a uniformly aggregated state.

Keywords: *CuO nanoparticles, urea, sucrose, solution combustion*

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1. Introduction

The Copper oxide have attracted the great deal of attention on basic as well as applied research community because of their optical, electrical, thermal properties. [3] CuO is applicable in various fields such as gas sensors, for magnetic storage media, in solar energy transformation of catalysis etc. [4]

In particular, the CuO nanoparticles show unique physicochemical properties due to its small particle size, high specific surface area and high surface activity. Recently it has been recognized as a promising candidate for application as organic synthesis catalyst, solid propellant combustion catalyst and blue color colorant for fireworks.

Different techniques are presented to develop CuO nanoparticles, which includes sol gel method, solvothermal, hydrothermal, alkoxide-based preparations [10-13], etc. However, these methods have disadvantages such as high cost of precursors, high temperature requirements, long time and high energy consumption.

Solution combustion synthesis (SCS) is the method for mass production of a variety of high purity nanoparticles with uniform and crystalline properties at relatively low temperatures (350-600 °C) in short time using exothermal redox reaction between fuel and oxidizer. Recently, there has been a growing interest in the world for the mass synthesis of various oxide nanoparticles by solution combustion synthesis and their application in many fields. [1-3]

In solution combustion synthesis, metal nitrates (oxidizers) and organic compounds (fuels) such as urea, glycine and citric acid are generally used as reaction precursors. [3-6]

The choice of fuel for the SCS is the most important factor.

In the solution combustion synthesis of CuO nanoparticles copper nitrate as an oxidizer and carbohydrazide, glycine, citric acid, urea, etc. as the fuel are used. [1-3] The cost of synthesis is high because all fuels are relatively expensive, except urea. The quality of the CuO nanoparticles formed is relatively low when urea is used as fuel.

In the present study, a solution combustion synthesis scheme of CuO nanoparticles using copper nitrate as an oxidant, urea as a fuel and sucrose as an additional fuel was first proposed, and then the optimum synthesis condition based on the analysis of the effect of fuel-to-oxidant ratio and synthesis temperature on the product properties was determined. Its characteristics were investigated.

2. Materials and methods

2.1. Chemicals

The pure chemicals, copper nitrate hexahydrate ($Cu(NO_3)_2 \cdot 6H_2O$), urea (CH_4N_2O), and sucrose ($C_{12}H_{22}O_{11}$), obtained from Sigma-aldrich with analytical grade were used for the synthesis.

2.2. Synthesis of CuO nanoparticles

The CuO nanoparticles were prepared by the solution combustion synthesis using $Cu(NO_3)_2 \cdot 6H_2O$ as the oxidant, urea as the fuel and sucrose as the additive fuel.

On the base of the propellant chemistry [38], the redox process occurring during the combustion is as follows:

$$3Cu(NO_3)_2+5CO(NH_2)_2 = 3CuO+5CO_2+8N_2+10H_2O$$

First, 40% of copper nitrate solution was prepared using copper nitrate [$Cu(NO_3)_2 \cdot 6H_2O$] of analytical grade. According to Eq. 1, a homogeneous mixture was prepared by adding a corresponding amount of urea (CH_4N_2O) to 10 g of copper nitrate solution, varying Φ_e to 0.1, 0.2 and 0.3, and stirring for 15 min. Sucrose ($C_{12}H_{22}O_{11}$) was added in the same amount as urea.

$$m_f = 60 \frac{O_V}{F_V} n_o \Phi_e \quad (1)$$

where m_f - amount of fuel (urea), g

O_V, F_V- oxidizer[$Cu(NO_3)_2$] and fuel's valency (respectively -10, +6)

n_o - amount of oxidizer, mol

Φ_e- fuel to oxidizer ratio

For 10 g of 40% copper nitrate solution, n_o = 0.0213 mol, and in Eq. 1, 60 is the molar mass of the fuel.

The mixture was placed in a preheated muffle furnace, after combustion reaction for 10 min, it was removed from the furnace and cooled to collect the powder. The synthesis temperature was varied to 400, 500 and 600 °C, and the temperature control of the furnace was realized using a PID-mode automatic temperature controller. The temperature control accuracy is ±5 °C at the corresponding holding temperature.

The solution was boiled and underwent dehydration, followed by decomposition with the evolution of the large amount of gases. At the end of the combustion reaction, the voluminous and fragile foam was produced.

2.3. X-ray diffraction (XRD)

Structural characterizations of the oxide samples were performed using the XRD Rigaku Smartlab (Cu k_α, λ=0. 154178 nm, step angle=0. 04°). The system was operated at 40 kV and 30 mA. Diffraction patterns were recorded in the 2θ range of 30°–70°. The crystallite size of the obtained powder was calculated using the Debye Scherrer formula [39]:

$$D = \frac{0.9 \times 竹}{汕 \times \cos 牟} \quad (2)$$

where D represents the crystallite size in nm, λ is the radiation wavelength (λ = 0.15406 nm), β denotes the full width at half of the maximum of the diffraction lines in radians, and θ represents the Bragg-angle. In addition, structural phase and crystallite size were determined by the Rietveld refinement analysis, using MDI Jade program.

2.4. Scanning electron microscopy (SEM)

The surface morphologies of the synthesized powder were observed using the JSM-6610A SEM instrument.

2.5. Brunauer–Emmett–Teller (BET) surface area measurements

The specific surface area of the oxides was determined by the BET nitrogen gas adsorption-desorption analysis conducted at 77 K using the JW-BK-22 instrument.

2.6. Scanning probe microscopy (SPM)

The scanning probe microscopy of the oxides was carried out using the CSPM-5500 instrument.

3. Results and discussion

3.1. Changes of in crystallite size and morphology of powder with Φ_e

To investigate the change in the crystallite size of CuO powder produced during solution combustion reaction, for the products obtained during combustion reaction of mixed solutions with Φ_e = 0.1, 0.2, and 0.3 at T = 600 °C X-ray diffraction analysis was conducted and the change in crystallite size with Φ_e was investigated.

Fig. 1 shows the X-ray diffraction pattern of the prepared samples. The XRD peak

position was consistent with the copper oxide and the sharp peaks with high intensity indicated the crystalline nature. The peaks were observed at 35.5 ° and 78.7 ° corresponding to ($\bar{1}11$) and (111) diffraction planes respectively of the monoclinic structure of CuO (JCPDS:80-1916). Peaks of impurities were not detected, indicating that the CuO nanoparticles are pure and well crystalline. These results shows that there is no significant change in the phase composition at Φ_e = 0.1-0.3.

The result of the crystallite size change with Φ_e using X-ray diffraction data was shown in Fig. 2.

As can be seen from the figure, the crystallite size increased with increase of Φ_e.

For applications of nano-powder, the particle size and the powder particle shape are often more important than the crystallite size.

The SEM images of the combustion products synthesized at different Φ_e were shown in Figure 3. Using the present SEM images, it is difficult to clearly identify the change of powder state with the change of Φ_e.

As shown in the figure 3, the powder obtained at Φ_e=0.1 had more aggregation than the powder obtained at Φ_e=0.2. It can be attributed to the slower combustion of lower fuel amount, which leads to more particle growth and aggregation.

It is clear that the condition of Φ_e= 0.2 is more reasonable.

3.2. Changes of particle size with temperature

SPM image of the solution combustion products synthesized at different temperatures was shown in Figure 4. As shown in Figure 4, the particles were basically spherical and the average particle sizes were respectively 127nm, 121nm, 104nm at 600℃, 500℃ and 400℃. This is because the higher the temperature, the more severe the grain growth.

Brunauer–Emmett–Teller (BET) surface area measurement result of the solution combustion products synthesized at different temperatures was shown in Fig 5.

The above results indicate that the combustion synthesis temperature required for CuO nanoparticles preparation is 400°C.

The results of the study show that the optimum solution combustion synthesis condition for CuO nanoparticles is Φ_e=0.2 and T=400°C. The specific surface area of the BET is 70.15m^2/g, D_{90}=150nm, and the powder remains uniformly aggregated.

4. Conclusion

CuO nanoparticles were synthesized by a novel method using mixed fuel using solution combustion reaction. Using copper nitrate as an oxidizer, urea as a fuel and sucrose as an additional fuel, the crystallite size of the combustion products, the aggregation state of the powder, the particle size and size distribution of the powder, and the specific surface area change were analyzed and the optimum combustion synthesis condition was determined.

It was found that the phase, particle size and specific surface area of CuO nanoparticles depend on the fuel-to-oxidizer ratio (Φ_e) and temperature. The optimum solution combustion synthesis condition for CuO nanoparticles is Φ_e=0.2 and T=400°C. The specific surface area of the BET is

$70.15 m^2/g$, D_{90}=150nm, and the powder remains uniformly aggregated.

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Figures caption

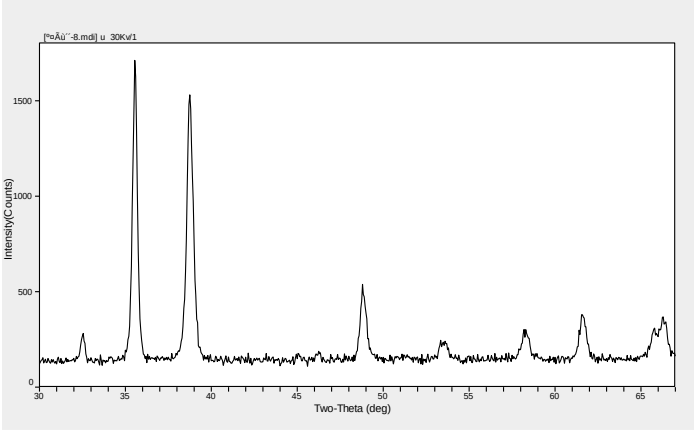
Figure 1. XRD patterns of the solution combustion products synthesized at different fuel-to-oxidant ratios (Φ_e = 0.1, 0.2, and 0.3).

Figure 2. Change of crystal size at different fuel-to-oxidant ratios (Φ_e = 0.1, 0.2, and 0.3).

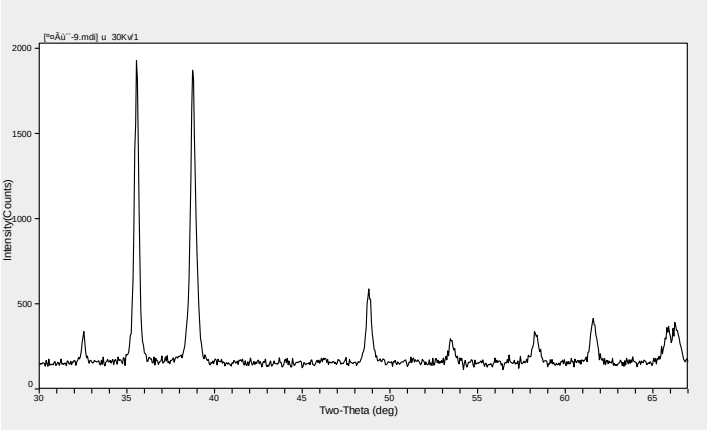
Figure 3. SEM images of the solution combustion products synthesized at different fuel-to-oxidant ratios (Φ_e = 0.1, 0.2, and 0.3).

Figure 4. SPM images of the solution combustion products synthesized at different temperatures (T = 600°C, 500°C, and 400°C).

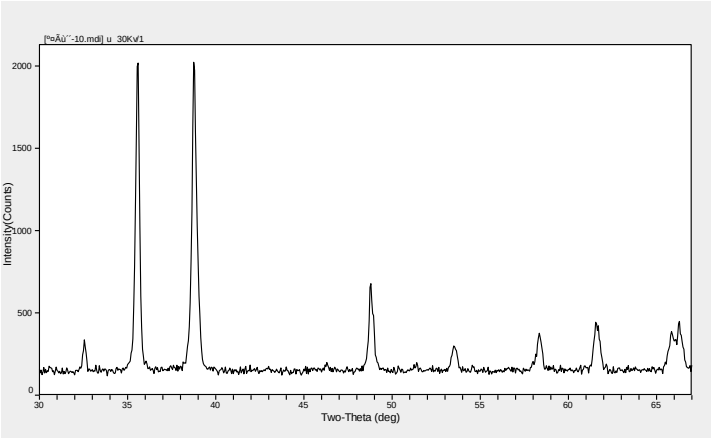
Figure 5. Brunauer-Emmett-Teller(BET) specific surface area measurement result.



Φ_e = 0.1



Φ_e = 0.2



$\Phi_e = 0.3$

Figure 1.

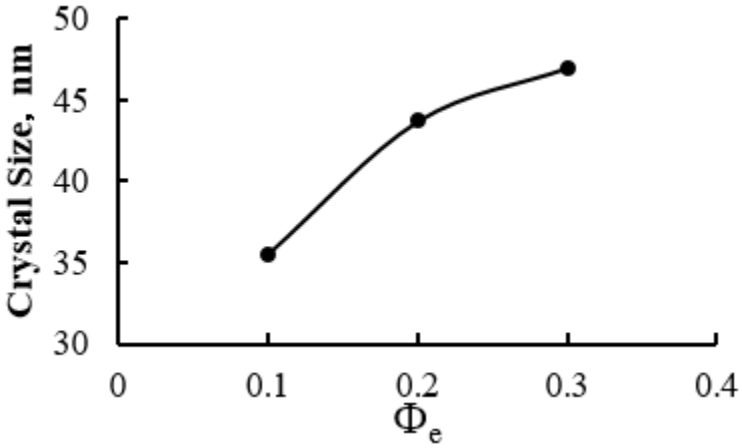
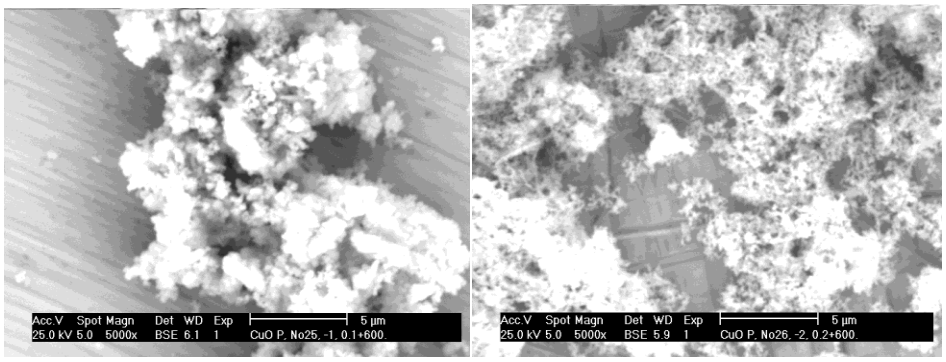
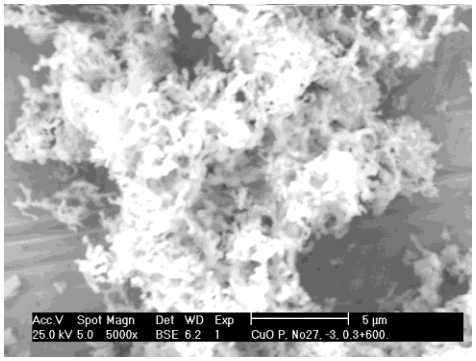


Figure 2.

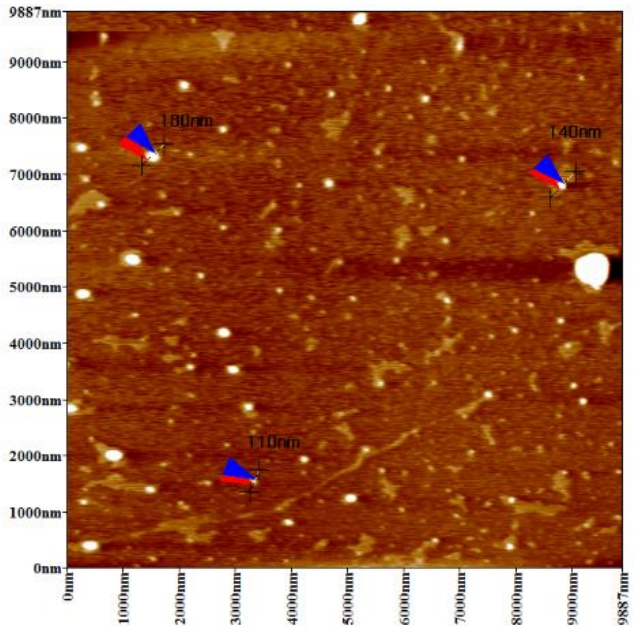


$\Phi_e = 0.1$ $\Phi_e = 0.2$



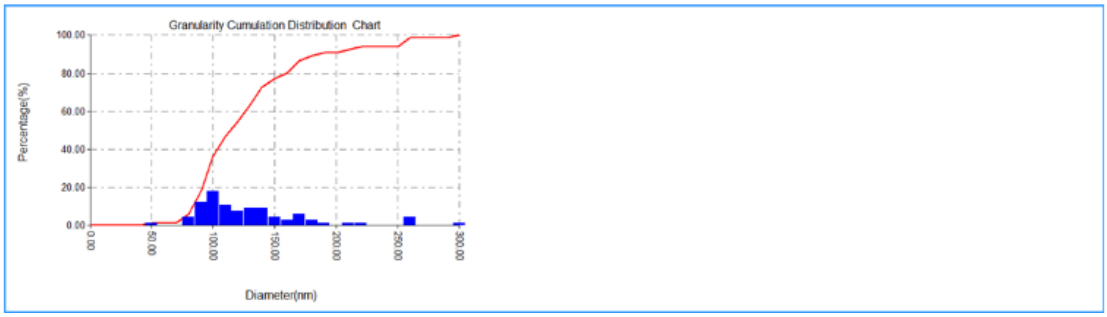
$\Phi_e = 0.3$

Figure 3.

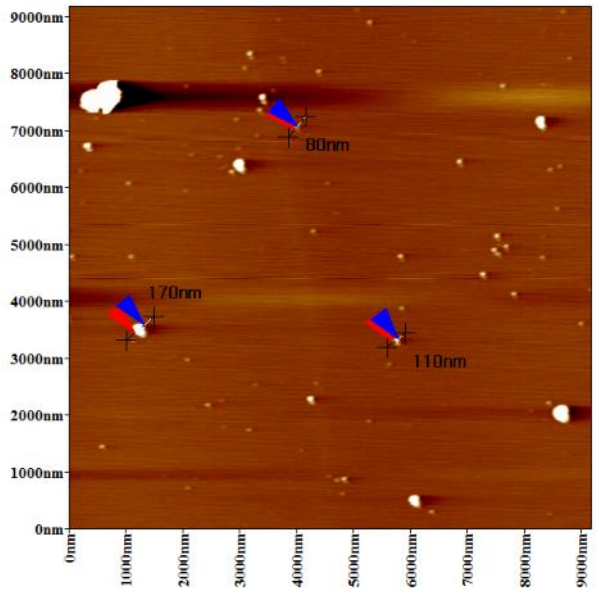


Avg. Diameter:127.07 nm <=10% Diameter:80.00 nm
<=50% Diameter:110.00 nm <=90% Diameter:180.00 nm

Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)
50.00	1.52	1.52	130.00	9.09	83.84	190.00	1.52	90.91
80.00	4.55	6.06	140.00	9.09	72.73	210.00	1.52	92.42
90.00	12.12	18.18	150.00	4.55	77.27	220.00	1.52	93.94
100.00	18.18	36.36	160.00	3.03	80.30	260.00	4.55	98.48
110.00	10.61	46.97	170.00	6.06	86.36	300.00	1.52	100.00
120.00	7.58	54.55	180.00	3.03	89.39			

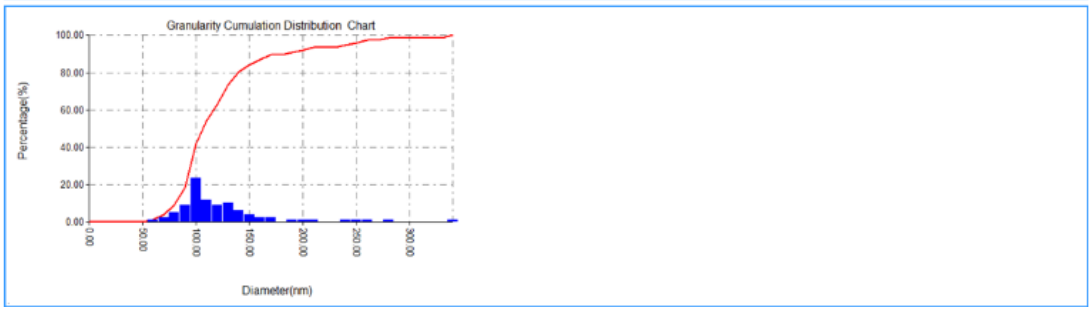


T = 600℃

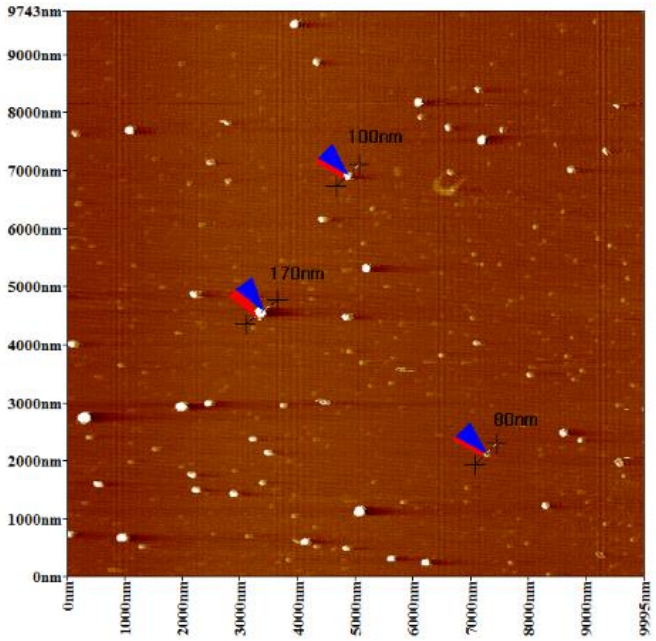


Avg. Diameter:121.25 nm
<=10% Diameter:80.00 nm
<=50% Diameter:100.00 nm
<=90% Diameter:170.00 nm

Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)
60.00	1.32	1.32	130.00	10.53	73.68	210.00	1.32	93.42
70.00	2.63	3.95	140.00	6.58	80.26	240.00	1.32	94.74
80.00	5.26	9.21	150.00	3.95	84.21	250.00	1.32	96.05
90.00	9.21	18.42	160.00	2.63	86.84	260.00	1.32	97.37
100.00	23.68	42.11	170.00	2.63	89.47	280.00	1.32	98.68
110.00	11.84	53.95	180.00	1.32	90.79	340.00	1.32	100.00
120.00	9.21	63.16	200.00	1.32	92.11			

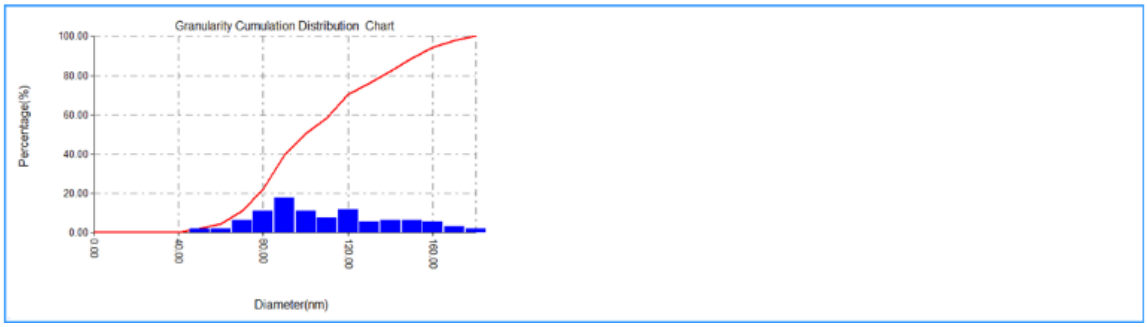


T = 500℃



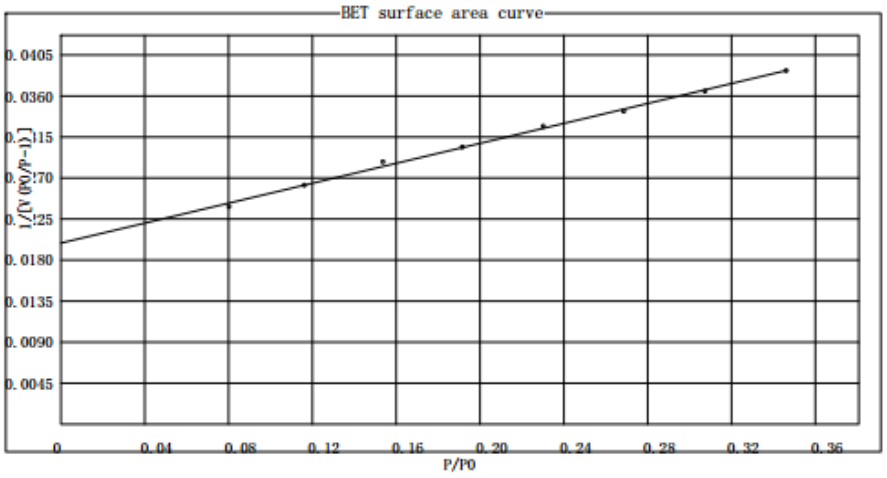
Avg. Diameter:104.87 nm
<=10% Diameter:60.00 nm
<=50% Diameter:90.00 nm
<=90% Diameter:150.00 nm

Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)
50.00	2.20	2.20	100.00	10.99	50.55	150.00	6.59	89.01
60.00	2.20	4.40	110.00	7.69	58.24	160.00	5.49	94.51
70.00	6.59	10.99	120.00	12.09	70.33	170.00	3.30	97.80
80.00	10.99	21.98	130.00	5.49	75.82	180.00	2.20	100.00
90.00	17.58	39.56	140.00	6.59	82.42			



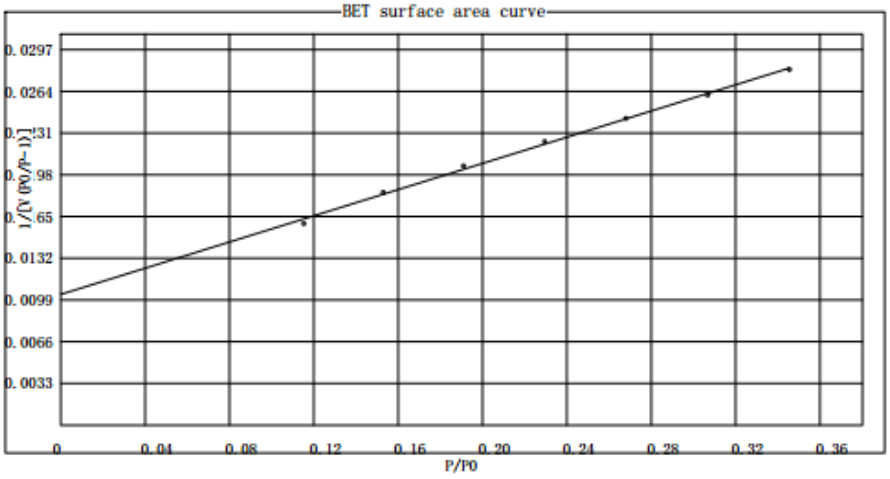
T = 400℃

Figure 4.



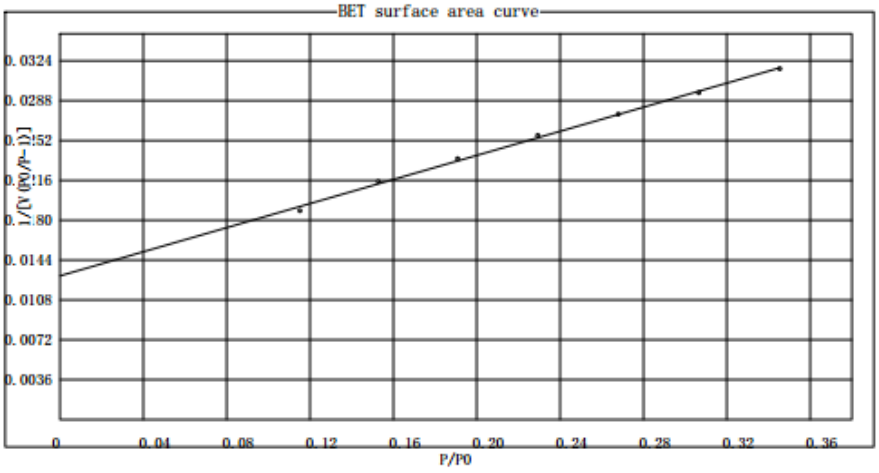
Slope	Intercept	Vm	specific surfac	C	Cc
0.05467	0.01987	13.41634	58.49526	3.75104	0.99851

T = 600℃



Slope	Intercept	Vm	specific surfac	C	Cc
0.05180	0.01035	16.09155	70.15917	6.00613	0.99858

T =500℃



Slope	Intercept	Vm	specific surfac	C	Cc
0.05441	0.01298	14.83916	64.69875	5.19347	0.99876

T = 400℃

Figure 5.

Table 1. BET specific surface area and average particle size of powder at different temperatures.

Temperature, ℃	BET specific surface area, m^2/g	Average particle size, nm
600	58.49	127
500	64.69	121
400	70.15	104