13

Single Step Solid Combustion Route for Preparing Nanosized NiO.

Rasha M. Abd El-Wahab, Doaa M. El-Mekkawi, S. A. Hassan[*] and M.M Selim[#]
*Physical Chemistry Department, National Research Center and *Chemistry Department, Faculty of Science, Ain Shams University, Cairo, Egypt.*

THE COMBUSTION synthesis technique using glycine and urea as fuels and nickel nitrate as an oxidizer is able to produce nano crystallite nickel oxide powder. The effects of fuel type, fuel-to-oxidizer ratios (F/O) and temperatures on phase composition and crystallite size of the combustion products were investigated. Thermogravimetric analyses (TGA) were performed to analyze the thermal behaviors of the starting materials. The products were analyzed using X-ray diffraction (XRD), Particle Size Analyzer (PSI), Transmission electron microscope (TEM) and surface area measurements. Optimization processes showed that the best F/O was 1:1 for glycine and 4:1 for urea. The best temperature was 400 °C to obtain crystallite size in nano scale, *i.e.* 13.4 nm for powder produced using urea with high surface area 83.8 m^2/g and 23.7 nm using glycine with surface area 40.6 m^2/g. On the other hand, the combustion process in absence of fuel is not efficient in preparing nano nickel oxide. Comparison with previous studies shows that our preparation method is efficient than the commonly used method.

Keywords: Combustion, Urea, Glycine, Nickel oxide and Nanoparticles.

The production of catalysts with high surface areas is still one of the challenging research topics in the field of surface and catalytic chemistry. However, in spite of the efforts and resources devoted to this subject, combustion synthesis represents a quick, straightforward preparation process to produce homogeneous, very fine, crystalline and unagglomerated oxide powders without the intermediate decomposition and/or calcining steps[1].

Combustion synthesis (CS) or self-propagating high-temperature synthesis (SHS) is an effective, low-cost method for production of various industrially useful materials. Today CS has become a very popular approach for preparation of nanomaterials and is practised in many countries. Recently, a number of important breakthroughs in this field have been made, notably for development of new catalysts and nanocarriers with properties better than those for similar traditional materials[2]. In CS, the exothermicity of the redox (reduction –oxidation

[#]Corresponding author E-mail: mmaselim@yahoo.com

or electron transfer) chemical reaction is used to produce useful materials. Depending upon the nature of reactants: elements or compounds (solid, liquid or gas); and the exothermicity (adiabatic temperature, *T)*. CS processes are characterised by high-temperatures, fast heating rates, short reaction times [1,3,4] and effective method for producing fine and nanosized oxide powders[5]. These features make CS an attractive method for the manufacture of technologically useful materials at lower costs[3,4,6,7] compared to conventional ceramic processes. Avgouropoulos *et al.* [8] reported that the catalyst obtained by combustion method was better than that obtained by co-precipitation, citrate-hydrothermal and impregnation methods. Some other advantages of CS are [9-13] : The method needs little heating to start a rapid reaction, the method requires little or no further processing such as calcinations or milling. There is a considerable savings in time, energy and equipment compared with the conventional techniques. Formation of high-purity products, stabilization of metastable phases, formation of virtually any size and shape products.

The process can be applied to prepare novel-oxides containing dopant elements, low-temperature compounds and cermets which are difficult to synthesize by the conventional methods.

The process uses the heat generated by the chemical reaction between fuel and metal nitrates to convert the metal ions to the target materials which are the key feature of this process, as the heat required to maintain the chemical reaction is supplied from the reaction itself (internal source) and not from an external source[14].

The process is based on the mixing of reactants that oxidize easily, such as nitrates, and an organic fuel, acting as a reducing agent. An external heat supply is needed to promote the ignition of the mixture leading to a self-sustaining and exothermic redox reaction[15].

As oxidizers, metal nitrates of the desired cation are preferred because they are water-soluble, contain nitrogen, and they usually melt at only few hundred degrees.

As fuels, molecules containing carboxylic and/or amine groups are mainly used, since they are a source of C, N and H, which form CO_2, NO_x and H_2O liberating heat. The combustion characteristics are closely related to the selection of fuel. A good fuel should react non-violently and act as a complexant for metal cations[4].

Urea (CH_4N_2O) is the most popular fuel because it is commercially available, cheap[17], produces high temperatures, generates low volume of gases[9,18]. The resulting product is usually a product that is dry, has high chemical homogeneity and purity, and is agglomerated into highly fluffy foam[19] by the escaping gases during the combustion reaction[20]. On the other hand, glycine ($C_2H_5NO_2$) is also used as a fuel, as it is a simple amino acids, known to act as a complexing agent for a number of metal ions, since it has a carboxylic acid group at one end and amino group at the other end. Such types of zwitterionic character of a glycine molecule

can effectively complex metal ions of varying ionic sizes, which helps in preventing their selective precipitation and maintaining compositional homogeneity among the constituents[4]·These fuels differ in the reduction potential and the amount of gases they generate, thus affecting the characteristics of the reaction products.

The present work reports and discusses the results of the effect of temperature, fuel concentration and fuel type on purity and crystal size of the economically synthesized nickel oxide catalyst by the combustion reaction.

Experimental

Materials

Urea (DELTA Co, Talkha Industry, 46%), glycine (99%), Rankem were used as fuels and nickel nitrate (Laboratory Rasayan, 98%) as oxidizer. Oxide samples were prepared by mixing calculated proportions of nickel nitrate with different amounts of urea and glycine.

Powder preparation

In our experiments, the ratios of fuel to nickel nitrate were (1:1, 2:1, 3:1, 4:1) in weight percent and heated at different temperatures (300°C, 400°C, 500°C). The mixed precursors were placed in a porcelain crucible and burn it in the furnace at these different temperatures. In all cases, the reaction is carried out in an open recipient; huge amounts of fumes were produced and, after ignition a fragile foam was formed that easily crumbled into powder.

To investigate the characteristics of combustion synthesized powders with these fuels, other powders were also prepared in the absence of fuels at temperatures 300-500°C.

Characterization

X-Ray Diffraction (XRD)

X-Ray diffractograms were obtained with The XRD-6000 series, including stress analysis, residual austenite quantitation, crystallite size / lattice strain, crystallinity calculation, materials analysis via overlaid X-ray diffraction patterns Shimadzu apparatus using nickel-filter and Cu-Kα target, Shimadzu Scientific Instruments (SSI).

The Debye–Scherrer, Eq. (1), was used to calculate the crystallite sizes of NiO from XRD data[16,17, 21-23].

$$d_{XRD} = 0.89\lambda / \beta\cos\theta \qquad (1)$$

where d_{XRD} is the volume average diameter of the crystallite, λ is the Cu Kα1 wavelength, β is the full width at half of the maximum in radians and θ is the Bragg-angle.

Weight fractions calulations: Weight fractions for the crystalline NiO in non pure samples can be calculated according to Eq. (2)[24]

$$X_1 = I_1 / I_1 + I_2 \qquad\qquad (2)$$

where X_1 is the weight fraction of crystalline NiO phase, I_1 is the intensity of major peak of NiO phase and I_2 is the intensity of major peak of Ni phase.

Thermogravimetric analyses (TGA)

The Pyris 6 TGA Thermogravimetric Analyzer from Perkin Elmer was used for thermogravimetric analyses.

Particle size analyzer (PSI)

The particle size of NiO samples were analyzed using Beckman Coulter - N5 Submicron particle size analyzer, USA.

Surface area analyzer

The surface area of NiO samples were analyzed using SA 3100 Beckman Coulter surface area analyzer, Miami, Florida.

Transmission electron microscope (TEM)

The TEM study was taken with JEOL- JEM-2100 HR TEM

Results and Discussion

Thermal analyses of the starting materials

TGA was performed to analyze the thermal behaviors of the precursors. It was measured with a heating rate of 10 °C min^{-1} under N_2 (g) purge between 50 and 600 °C. Figure1 shows TGA of glycine, urea and nickel nitrate. Urea shows four weight loss stages. The first stage with a sharp weight loss of 73.7% takes place in the temperature range of 145–230°C. The second loss is 8% in the temperature range of 230–257 °C. The third loss is 14 % in the temperature range of 257 to 330 °C and the fourth loss is 3.5% in the temperature range of 330 to 600 °C. The overall weight loss measured at 600 °C is 98.8%. Glycine curve shows three weight loss stages. The first stage with a sharp weight loss of 54.6% takes place in the temperature range of 228–286 °C, which is followed by two slow weight losing stages in 286 to 425°C and 425 to 600 °C. The overall weight loss measured at 600 °C is 77%. Nickel nitrate curve shows complete decomposition at 375 °C with overall loss about 72.3%, this seems to be related to the formation of NiO. From TGA curves we can recognize that glycine and urea are suitable fuels for nickel nitrate in the combustion process as the ignition point of these fuels associated with the sharp loss is lower than 375 °C.
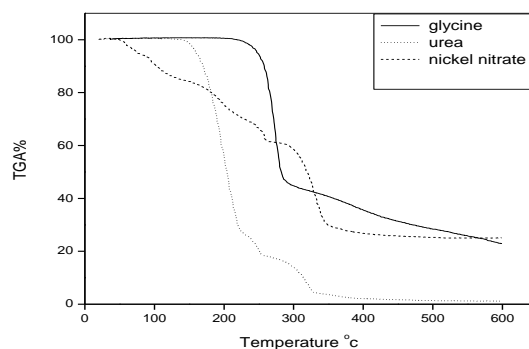
Fig. 1. TGA of glycine, urea and nickel nitrate.

Phase formation with fuel type at different temperature

Figure 2 shows the X- ray diffractogram of Ni: NiO samples synthesized from F/O 1:1 of urea to nickel nitrate at different temperatures. Weight fractions for the crystalline NiO phase were found to be (35.11, 28.48 and 68%) at 300, 400 and 500 °C, respectively. It reveals that at 300 and 400 °C the crystalline Ni phase presents as a major phase while at 500 °C the crystalline NiO phase presents as the major phase. Figure 3 shows that the crystalline NiO can be obtained as a major with weight fraction 61.86% at 300 °C and as a pure phase at 400 and 500 °C by using F/O 1:1 of glycine and nickel nitrate. All diffraction peaks can be well indexed to the pure cubic phase of NiO (JCPDS card, no. 78-0643). No other impurity peaks were observed in the XRD patterns, indicating that the precursor has been completely converted to the NiO at 400 and 500 °C. This observation seems to be related to fuels composition, as the fuels consisted of both the carboxylic acid group and amine group like glycine were strongly reacted with metal nitrates. The reactivity of this system was strongly dependent on the composition of fuels[14]. The reaction of 300°C with urea leads to the formation of brown residue, *i.e.* uncompleted combustion of urea. This may explain the formation of only metallic nickel without formation of nickel oxide, *i.e.* the brown product protects the formed metal from oxidation.
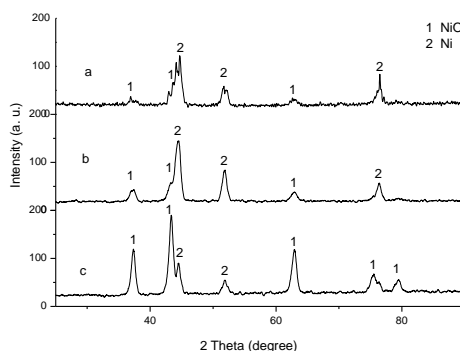

Fig. 2. XRD patterns as-prepared Ni and NiO: (a) 300 °C, (b) 400 °C and (c) 500 °C with equal ratios of urea: nickel nitrate.
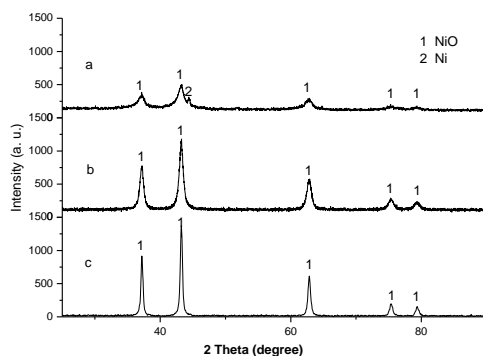
Fig. 3. XRD patterns as-prepared Ni and NiO: (a) 300 °C, (b) 400 °C and (c) 500 °C with equal ratios of glycine: nickel nitrate.

Phase formation with different ratios of fuel to nitrate at different temperature

It is known that one of the major parameters that determine the phase formation of the synthesized powder is F/O in the combustion reaction[14]. Figure 4 compares the XRD pattern of samples a and b to investigate the effect of increasing urea ratio at 300°C. Sample (a) shows the formation of low crystalline phase of nickel metal as a major but there was minor evidence of the presence of nickel oxide. Sample b leads to an amorphous phase indicating in complete combustion reaction[18]. Raising the temperature to 400°C (Fig. 5) indicates that combustion took place and crystalline phases were registered in the XRD pattern and crystalline phase of NiO was obtained at F/O 2:1 and 4:1of urea to nickel nitrate with crystal size 19.4 and 13.4 nm, respectively as shown in Table 1 which is determined by XRD line broadening analysis. All diffraction peaks can be well indexed to the pure cubic phase of NiO (JCPDS card, no. 78-0643). At lower ratios crystalline phase of Ni was obtained. Increasing the temperature to 500°C as shown in Fig. 6 leads to pure crystalline NiO formation with all ratios with small appearance of Ni. The XRD patterns become sharp with increasing F/O above 1:1 this shows that the crystallite sizes become larger. The crystallite size of the powders was calculated by the Scherrer formula, and the results are given in Table1.
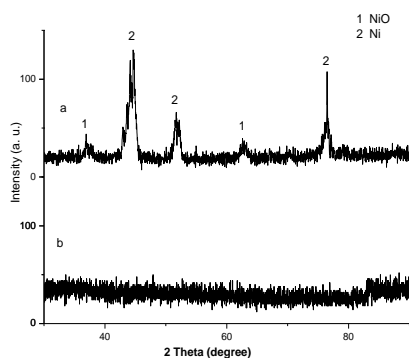

Fig. 4. XRD patterns of the as-prepared when burning urea: nickel nitrate with F/O1:1 (a), 2:1 (b) 300°C.
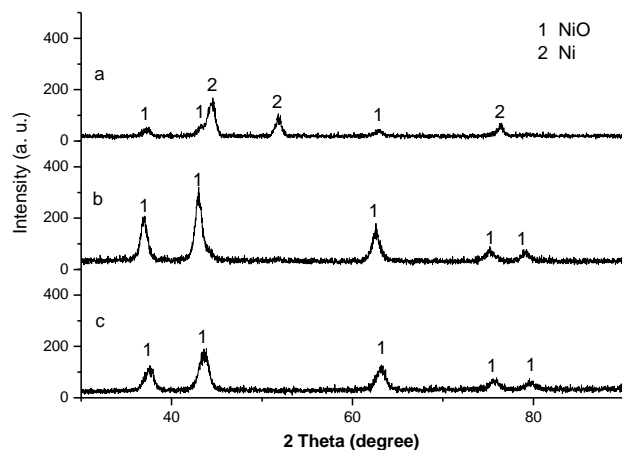
Fig. 5. XRD patterns as-prepared Ni and NiO at 400 °C with F/O 1:1 (a), 2:1 (b), and 4:1 (c) F/O of urea: nickel nitrate.

TABLE. 1. Effect of fuel type,fuel ratio and temp. on crystal size.

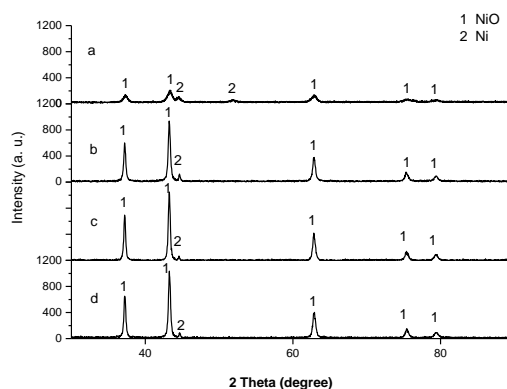| Fuel	Temp°C	F/O	Crystal size (nm)	NiO formation (%)
Glycine	300	1:1	15.3	61.45
		2:1	-	amorphous
		3:1	-	amorphous
		4:1	-	amorphous
	400	1:1	23.5	100
		2:1	23.7	100
		3:1	38.2	100
		4:1	186.3	100
	500	1:1	186.3	100
		2:1	186.3	100
		3:1	181.4	100
		4:1	186.3	100
Urea	300	1:1	32.8	35.11
		2:1	-	amorphous
		3:1	-	amorphous
		4:1	-	amorphous
	400	1:1	17.4	27.5
		2:1	19.4	100
		4:1	13.4	100
	500	1:1	23.5	67.23
		2:1	189.9	100
		3:1	189.9	100
		4:1	189.9	100

Fig. 6. XRD patterns as-prepared Ni and NiO at 500 °C with F/O 1:1 (a), 2:1 (b), 3:1(c)and 4:1(d) ratios of urea: nickel nitrate.

Figure 7 compares the XRD pattern of samples a and b to investigate the effect of increasing glycine ratio at 300°C which exhibit the same trend as in urea. The only difference is in sample (a) which shows the formation of crystalline phase of nickel oxide as a major with a minor formation of nickel, also sample b leads to an amorphous phase indicating in complete combustion reaction.
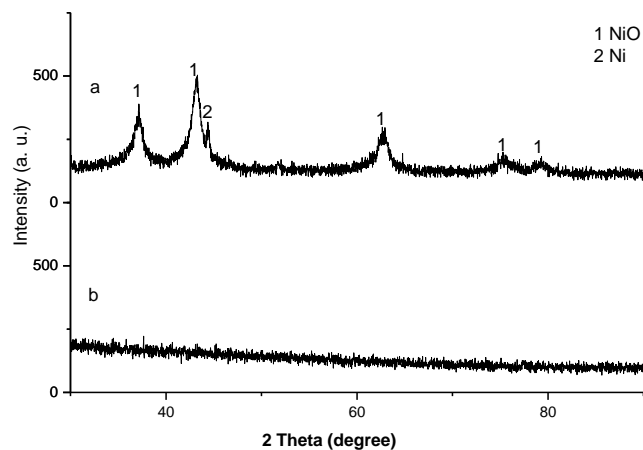

Fig. 7. XRD patterns as-prepared Ni and NiO at 300 C° with F/O 1:1 (a) and 2:1 (b) of glycine: nickel nitrate.

Figures 8 and 9 show that NiO can be prepared as pure crystalline phase at temperatures 400 and 500°C with the mentioned ratios of glycine. All diffraction peaks can be well indexed to the pure cubic phase of NiO (JCPDS card, no. 78-0643). The crystallite size of the powders obtained with F/O 1:1 is slightly smaller than that for ratio of 2:1[23], *i.e.* 23.4 and 23.7 nm can be obtained with F/O of 1:1and 2:1 of glycine to nitrate 400°C, respectively. The

F/O above 2 lead to a large increase in the crystallite size of the powders obtained. At 500°C the crystallite sizes are obviously very large with all the ratios. This may be mainly due to the high temperature during the glycine-nitrate combustion reaction, resulting in a substantial grain growth[23].
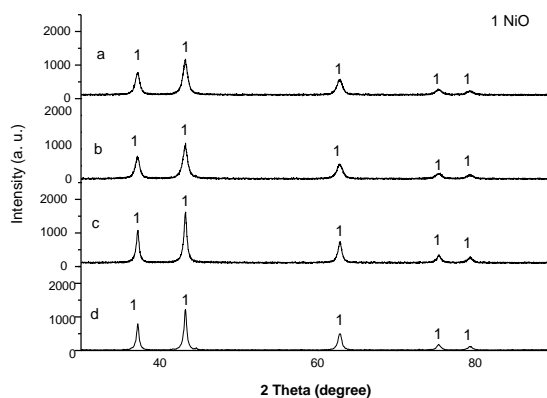

Fig. 8. XRD patterns of as-prepared Ni and NiO at 400 °C with F/O 1:1 (a), 2:1 (b), 3:1(c) and 4:1 (d) of glycine: nickel nitrate.
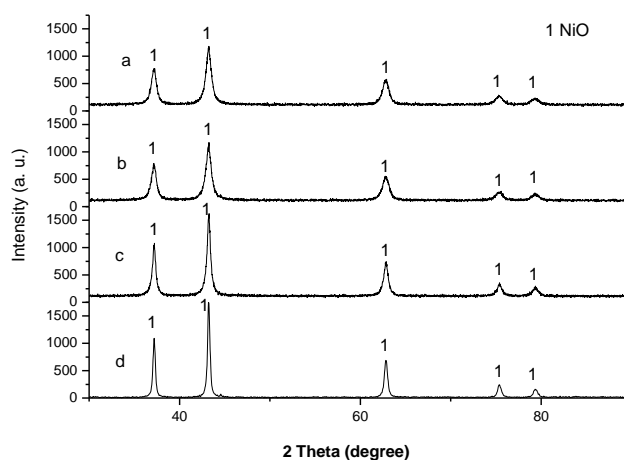

Fig. 9. XRD patterns of as-prepared Ni and NiO at 500 °C with F/O of glycine: nickel nitrate 1:1 (a), 2:1 (b), 3:1(c) and 4:1(d).

Figure 10 shows the combustion process of nickel nitrate in the absence of fuel at 300 °C nickel oxide and other phases can be found. Increasing the temperature to 400 and 500°C leads to the formation of nickel oxide. This process is not efficient not only in preparation of nano nickel oxide, but also in obtaining varieties of sizes. As shown in Table 2 the size does not change by increasing the temperature. At temperature below 300°C pure NiO cannot be

obtained as we still have peaks characteristic to Ni and Ni (OH) $_2$ at 300°C. It is expected that increasing temperature above 500°C will not lead to formation of nano sized nickel oxide since raising the calcining temperature and prolonging the soak time make the crystalline grains grow larger in size and weaken the reactivity[6], so obtaining pure nano sized particles in absence of fuel is difficult. On the other hand, combustion with fuels results in formation of virtually any size and shape pure products[9].

TABLE 2. Crystal sizes of combusted Nickel nitrate at different temperatures.

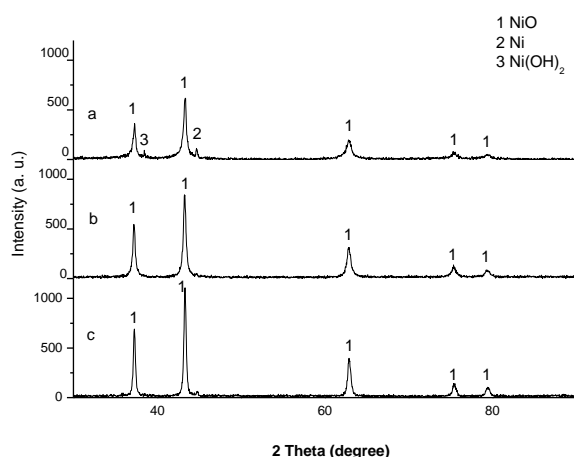| Temperature °C	Crystal size(nm)
300	189.9
400	189.9
500	189.9

Fig. 10. XRD patterns of combusted Nickel nitrate at 300(a), 400(b) and 500 °C (c).

Particle size and surface area measurements

Table 1 shows the effect of temperature, feul type and fuel to oxidizer ratio on the crystallite size of obtained phase. At 400 °C, a crystallite size of 13.4 and 23.5 nm can be obtained using urea and glycine, respectively with F/O 1:1 of glycine to nitrate and 4:1 urea to nitrate. On the other hand, from the particle size analysis, we can reveal that we succeeded to obtain particle size in nano scale as shown in Table 3 we obtain particle size of 6.2nm when using urea as a fuel. The surface area of the prepared NiO nanoparticles at the same F/O 2:1 for glycine and urea at 400 which determined to be found higher in case of glycine (40.6 m^2/g) than of urea (32.6m^2/g). Increasing the ratio of F/O in case of urea to 4:1 leads to decrease the crystallite size as shown in Table 1 which results in increasing the surface are a to be 83.8 m^2/g. On the other hand, as a result of the large increase in the crystallite size of the synthesized materialsa at F/O 4:1

using glycine, the corresponding specific surface area is expected to be drastically reduced[25]. Compared with the reported crystallite size ,surface area and weight fraction of NiO nanoparticles prepared by different methods, solution-phase[26], solution combustion[25,27], solvothermal[28] and microwave combustion[27],our sample synthesized at 400 ∘C with F/O 4:1 using urea is much enhanced as shown in Table 4.

Therefore, it is believed that the solid combustion is more efficient method in preparation of NiO nanomaterials with high surface area which enhance the catalytic properties than other mehods.

TABLE. 3. Surface area of the prepared powder with different conditions.

Fuel	F/O	Surface area (m^2/g)	Mean paticle size (nm)	Temperature °C
Urea	2	32.6	6.2	400
urea	4	83.8	-	400
glycine	2	40.6	111.8	400

TABLE. 4. Comparison of the crystallite size, surface area and weight fraction of NiO synthesized in this work with those of NiO reported in the literature.

Method	Crystallite size (nm)	Surface area (m^2/g)	Weight fraction (%)	Ref.
Solid combustion	13.4	83.8	100	this study
solution-phase	-	25.3	100	25
Solution combustion	7	99.7	68	24
solvothermal	25	-	not pure	27
Microwave combustion	20	73.5	not pure	26
Solution combustion	65	32.6	100	26

Transmission electron microscope (TEM) studies

Figure 11 represents the different morphologies of the as-synthesized NiO nanoparticles using different fuels with different F/O. The shapes of the as-synthesized NiO nanoparticle in Fig.11a show the spherical shape of NiO nanoparticles. Figure 11b shows the cubic shape of NiO nanoparticles with crystallite size of 10nm. Rapid evolution of large volume of the gaseous products during combustion dissipates the heat of the process and limits temperature increase, thus reducing the possibility of premature local partial sintering among the primary particles. The gas evolution also helps in limiting the interparticle contact resulting in a more easily friable product[29], so the increas of urea content increases the friability of the product. As increasing the urea increases the amount of energy release resulting in the change of the produced compound from spherical (F/O= 2) with particle size 20-40 nm to

tetragonal- like form (F/O=4) with particle size of 45 nm in length and 22 nm in width. As shown in Fig. 11a and Fig. 11c, respectively. So the fuel type and ratio affect the size and morphology of the obtained powders.
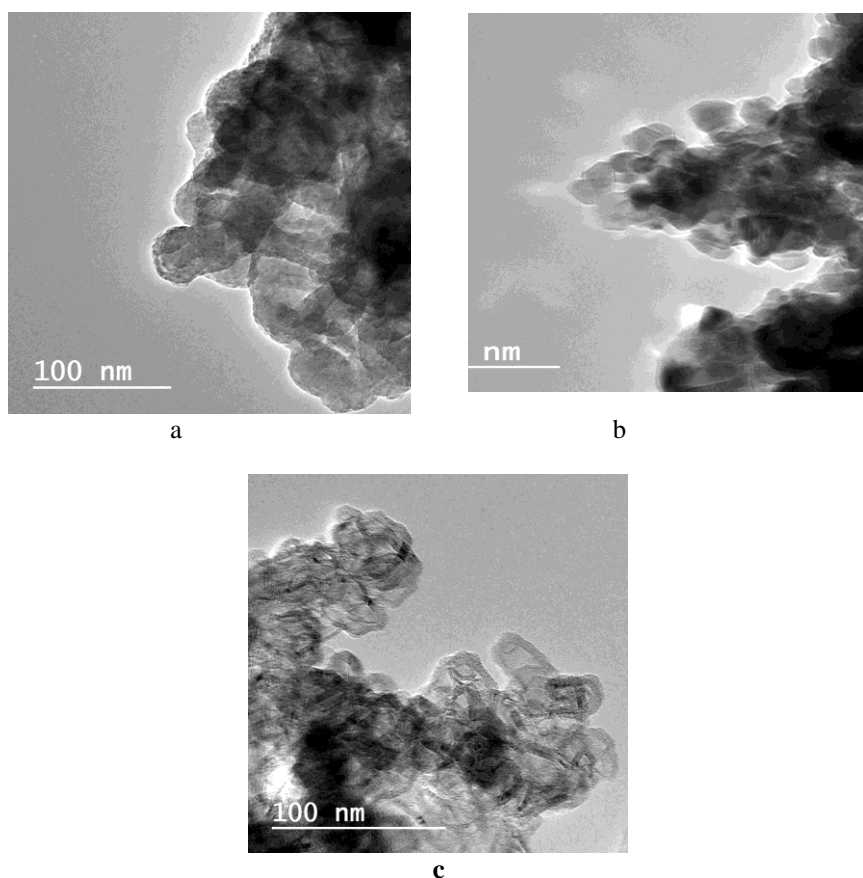

a   b

c

Fig. 11. TEM of the powder prepared at 400°C with F/O=2 with urea (a), F/O=2 with glycine (b) and F/O=4 with urea (c).

Conclusion

In the current investigation, the role of different fuel to oxidizer ratio, fuel type and temperature on synthesis condition of nano sized NiO were studied. The results are remarked as bellow:
• Nano crystallite nickel oxide can be prepared as a pure phase by combustion of nickel nitrate as oxidizer and glycine and urea as fuels in a single step.
• Optimization processes investigate that the best F/O was 4:1 for urea and 1:1 for glycine, the best temperature was 400 °C to obtain crystallite size in nano scale, *i.e.* 13.4nm for urea and 23.5nm. High surface area 83.8 m^2/g and 40.6 m^2/g were obtained for F/O=4 with urea and F/O=2 with glycin, respectively.

- Our optimized sample synthesized using urea is much enhanced when compared with NiO nanoparticles prepared by different methods.
- The combustion process in absence of fuel is not efficient not only in preparing nano nano nickel oxide, but also in obtaining varieties of sizes.

References

1. **Abu-Zied, B.M.,** Preparation of cadmium chromite spinel: a combustion approach. *Colloids and Surfaces A: Physicochem. Eng. Aspects*, **211**, 27 – 42 (2002).

2. **Aruna, S.T. and Mukasyan, A.S.,** Combustion synthesis and nanomaterials. *Current Opinion in Solid State and Materials Science*, **12**, 44–50 (2008).

3. **Alves, C.T., de Oliveira, A.S., Carneiro, S.A.V., Santos, R.C.D., Vieira de Melo, S.A. B., Andrade, H.M.C., Marques, F.C. and Torres, E.A.,** Transesterification of waste frying oils using $ZnAl_2O_4$ as heterogeneous catalyst. *Procedia Engineering*, **42**, 1928 – 1945 (2012).

4. **Xiaoming Guo, Dongsen Maob, Guanzhong Lu, Song Wang and Guisheng Wu,** Glycine–nitrate combustion synthesis of CuO–ZnO–ZrO_2 catalysts for methanol synthesis from CO_2 hydrogenation. *Journal of Catalysis*, **271**,178–185 (2010).

5. **Zhuravlev, V.D., Bamburov, V.G., Beketov, A.R., Perelyaeva, L.A., Baklanova, I.V., Sivtsova, O.V., Vasil'ev, V.G., Vladimirova, E.V., Shevchenko, V.G. and Grigorov, I.G.,** Solution combustion synthesis of α-Al_2O_3 using urea. *Ceramics International*, **39**, 1379–1384 (2013).

6. **Xiwei, Qi., Zhou, Ji. ZhenxingYue, Zhilun Gui and Longtu Li,** A simple way to prepare nanosized $LaFeO_3$ powders at room temperature. *Ceramics International,* **29**, 347–34.

7. **Won, C.W., Nersisyan, H.H., Won, H.I., Jeon, D.Y. and Han, J.Y.** Combustion synthesis and photoluminescence of $ZnS:Mn^{+2}$ particles. *Journal of Luminescence* **130**, 1026–1031 (2010).

8. **Avgouropoulos, G., Ioannides, T. and Matralis, H.,** Influence of the preparation method on the performance of CuO–CeO_2 catalysts for the selective oxidation of CO. *Applied Catalysis B: Environmental*, **56**, 87–93 (2005).

9. **Patil Kashinath, C., Aruna, S.T. and Mimani Tanu**, Combustion synthesis: An update, *Current Opinion in Solid State and Materials Science*, **6**, 507–512 (2002).

10. **Deraz, N.M. and Shaban, S.,** Optimization of catalytic, surface and magnetic properties of nanocrystalline manganese ferrite. *J. Anal. Appl. Pyrolysis*, **86**, 173–179 (2009).

11. **Cannas, C., Falqui, A., Musinu, A., Peddis, D., and Piccaluga,G**., CoFe$_2$O$_4$ nanocrystalline powders prepared by citrate-gel methods: Synthesis, structure and magnetic properties. *Journal of Nanoparticle Research*, **8**, 255–267 (2006).

12. **Deraz, N.M.,** Glycine-assisted fabrication of nanocrystalline cobalt ferrite system. *Journal of Analytical and Applied Pyrolysis*, **88**, 103–109 (2010).

13. **Zhenxing Yue, Ji Zhou, Longtu Li, Hongguo Zhang and Zhilun Gui**, Synthesis of nanocrystalline NiCuZn ferrite powders by sol-gel auto-combustion method. *Journal of Magnetism and Magnetic Materials*, **208**, 55-60 (2000).

14. **Jung, C.H., Park, J.Y., Oh ,S.J., Park, H.K., Kim, Y.S., Kim, D.K. and Kim, J.H.,** Synthesis of Li TiO ceramic breeder powders by the combustion process. *Journal of Nuclear Materials*, **253**, 203–212 (1998).

15. **Long Yulin,Yang Jun, Li Xiaoci, Huang Weiya, Tang Yu and Zhang Yuanming**, Combustion synthesis and stability of nanocrystalline La$_2$O$_3$ via ethanolamine-nitrate process. *Journal of Rare Earths*. **30**, 48 (2012).

16. **Patterson, A. L**., The Scherrer formula for X-ray particle size determination. *Phys. Rev.* **56** (10), 978–982 (1939).

17. **Nasiri, H., Bahrami Motlagh, E., Vahdati Khaki, J. and Zebarjad, S.M**., Role of fuel/oxidizer ratio on the synthesis conditions of Cu–Al$_2$O$_3$ nanocomposite prepared through solution combustion synthesis. *Materials Research Bulletin*, **47**, 3676–3680 (2012).

18. **Burgos-Montes, O., Moreno, R., Colomer, M.T. and Fari˜nas, J.C.,** Influence of combustion aids on suspension combustion synthesis of mullite powders. *Journal of the European Ceramic Society*, **26**, 3365–3372 (2006).

19. **Costa, A.C.F.M., Lula, R.T., Kiminami, R.H.G.A., Gama, L.F.V., De Jesus, A.A. and Andrade H.M.C.,** Preparation of nanostructured NiFe$_2$O$_4$ catalsts combustion reaction. *J. Mater Sci.* **41**, 4871-4875 (2006).

20. **Yen-Chun Liu and Yen-Pei Fu,** Magnetic and catalytic properties of copper ferrite nanopowders prepared by a microwave-induced combustion process. *Ceramics International*, **36**, 1597–1601 (2010).

21. **Yazhong Chen, Wei Zhou, Zongping Shao and Nanping Xu**, Nickel catalyst prepared via glycine nitrate process for partial oxidation of methane to syngas. *Catalysis Communications*, **9**, 1418–1425 (2008).

22. **Qigen Wang, Ranran Peng, Changrong Xia, Wei Zhu and Huanting Wang,** Characteristics of YSZ synthesized with a glycine-nitrate process. *Ceramics International*, **34**, 1773–1778 (2008).

23. **Tianmin He, Qiang He and Nan Wang**, Synthesis of nano-sized YSZ powders from glycine-nitrate process and optimization of their properties. *Journal of Alloys and Compounds,* **396**, 309–315 (2005).

24. **Garvie Ronald, C. and Nicholson Patrick, S.,** Phase analysis in zirconia systems. *Journal of American Ceramic Society*, **55**, 303-305 (1972).

25. **Pranati Sahoo, Dinesh, K. Misra, Jim Salvador, Julien, P.A. Makongo, Girija S. Chaubey, Nathan, J. Takas, John, B. Wiley and Pierre F.P.Poudeu**, Microstructure and thermal conductivity of surfactant-free NiO nanostructures. *Journal of Solid State Chemistry,* **190**, 29–35 (2012).

26. **Li-Na Jin, Qing Liu and Wei-Yin Sun**, Room temperature solution-phase synthesis of flower-like nanostructures of [$Ni_3(BTC)_2.12H_2O$] and their conversion to porous NiO. *Chinese Chemical Letters,* **24**, 663-667 (2013).

27. **Manikandan, A., Vijaya, J. Judith and Kennedy, L. John,** Comparative investigation of NiO nano- and microstructures for structural, optical and magnetic properties. *Physica E* **49**, 117–123 (2013).

28. **Anandan, K. and Rajendran, V.,** Morphological and size effects of NiO nanoparticles via solvothermal process and their optical properties. *Materials Science in Semiconductor Processing,* **14**, 43–47 (2011).

29. **Toniolo, J. C., Lima, M. D., Takimi, A. S. and Bergmann, C. P.,** Synthesis of alumina powders by the glycine–nitrate combustion process. *Materials Research Bulletin*, **40**, 561–571 (2005).

طريقة الخطوة الواحدة لاحتراق المادة الصلبة لتحضير اكسيد النيكل النانومترى

رشا عبد الوهاب ، دعاء المكاوى ، صلاح الدين عبده حسن* ومحمد محمد سليم

قسم الكيمياء الفيزيقية – المركز القومى للبحوث و*قسم الكيمياء – كلية العلوم – جامعة عين شمس – القاهرة – مصر.

تقنية الاحتراق باستخدام الجلايسين واليوريا كوقود ونترات النيكل كعامل مؤكسد قادرة على إنتاج مسحوق بلوري من أكسيدالنيكل النانو. فى هذا البحث درس تاثيرنوع الوقود، نسب الوقود الى العا مل المؤكسد ودرجات الحرارةعلى تركيب وحجم الكريستال الناتج من الاحتراق للأكسدة. تم إجراء التحليل الحراري الوزنى لتحليل السلوكيات الحرارية للمواد الأولية. وقد تم تحليل نواتج الاحتراق باستخدام حيودالأشعة السينية، ومحلل حجم الجسيمات ، والميكروسكوب الالكتروني النافذ وقياسات مساحة السطح. وأظهرت العمليات أن أفضل F/ O كانت1:1 للجلايسين و 1:4 لليوريا. وكانت أفضل درجة حرارة 400 درجة مئوية للحصول على حجم الكريستال في مقياس النانوأي 13,4 نانومترللمسحوق المنتج باستخدام اليوريا مع مساحة سطح عالية 83,8 متر^2/جرام و 23.7 نانومتر باستخدام الجلايسين مع 40,6 متر^2/جرام . من ناحية أخرى، فإن عملية الاحتراق فى غياب الوقود ليست فعالة في إعدادأكسيد النيكل النانو. وبالمقارنة مع الدراسات السابقة تبين أن الطريقة الحالية فعالة عن الأساليب المستخدمة بشكل شائع.