
Preparation of nanocompounds materials by combustion method using urea as fuel

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Abstract: LiNiCoO₂ (LCNO) has been prepared using combustion method with urea as the igniter and glycerol as the binding material due to its high activity. Urea, used as fertiliser, can be produced by way of sequence chemical approaches by means of the usage of H₂, N₂ and CO₂. It is good performance hydrogen and carbon dioxide as storage medium due to high energy density (16.9 M*J *L⁻¹, 10.1 weight percent of H₂) for direct urea fuel cell (DUFC). Nitrates of Li⁺, Co⁺² and Ni⁺² had been combined together to shape a homogenous medium. Required quantities of urea and glycerol have been brought to combination and made a good paste. This paste is heated to 400°C for 2h. The as-synthesised fabric used to be subjected to thermo-gravimetric (TGA) analysis to decide the top of the line varies of annealing temperatures.

Keywords: nanoparticle; LiCoNiO₂; direct urea fuel cell; DUFC; FTIR; X-ray diffraction; characterisation.

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1 Introduction

Among the compounds LiNiCoO_2 , Li-ion batteries are one a suitable compromise. In this work, to overcome the negatives produced by LiCoO_2 , LiNiO_2 and LiMnO_2 batteries, the new cathode material LiNiCoO_2 was manufactured using urea as a fuel and glycerol as a bond material (Haider et al., 2018a). Metal oxides can undertake a giant range of structural geometries with a digital shape that might also exhibit metallic, semiconductor, or insulator characteristics, endowing them with numerous chemical and bodily homes. They are particularly sol-gel (Christian et al., 2016; Haider et al., 2018b, 2018c), co-precipitation, combustion (Xu et al., 2015), pyrolysis (Feng et al., 2012), polyol (Al-Douri et al., 2012), etc. These techniques have been used put together nano-structured metallic oxides utilised to electrodes for Li-ion batteries (Jameel et al., 2016). The chemical characteristic of cathode substances fundamentally up on the type of the structure, orientation of the planes and morphology of the substances attribute to various synthesis process. A great challenge of the world storage energy industry today is to take action to slow global warming. While it is recognised that the earth's climate varies naturally, the majority of scientists now believe that greenhouse gas emissions attributable to human activities have been increased. Rapid increase in atmospheric CO_2 concentration in the atmosphere during last centuries is due to the effects of human activities. It is worth mentioning that three quarters of the gas emissions are attributable to burning fossil fuels.

In the present, the project, a multiplied practice of LCNO combustion method technique in air is observed and involves exoth. Redox response of an oxidiser (metal nitrate) and biological gas (urea and glycerol) to reap the preferred layered phase, annealing temperature, and were determined via means of XRD and FESEM to addition of FTIR analysis.

2 Experimental details

2.1 *Manufacture of NP's LiNiCoO_2*

LiNiCoO_2 used to be prepared through combustion strategy. The stoichiometric amount of LiNO_3 two (14%) and nickel nitrate $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (26.60%) additionally $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (29.57%) have been supplied from Harris Chemicals Corporation in England at high purity approximately (99.99%), taken along with urea as fuel, glycerol as binding manufacture with synthetic in a uniformity glue.

Above mixtures was once dissolved in adequate amount of de-ionised water 25 ml and put it in hot plate (magnetic stirrer) for 15 minutes at the temperature room two then affirm dissolution of the nitrates. The answer is then heated on hot plate heater to 100°C

for 30 min. Urea at excessive concentration with so little low awareness of two glycerol two introduced to the mixture two that was once homogenised as shown in Figure 1.

Figure 1 Block diagram of LiCoNiO₂ manufacture by combustion reaction

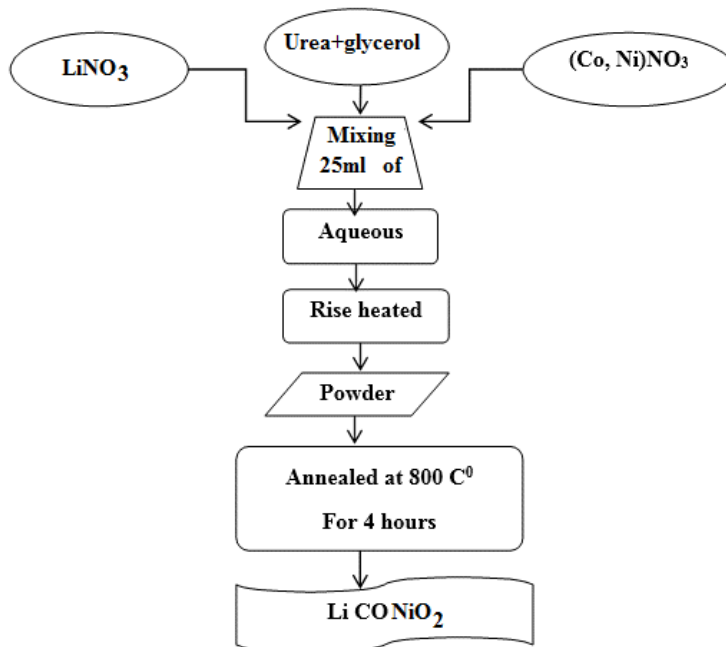
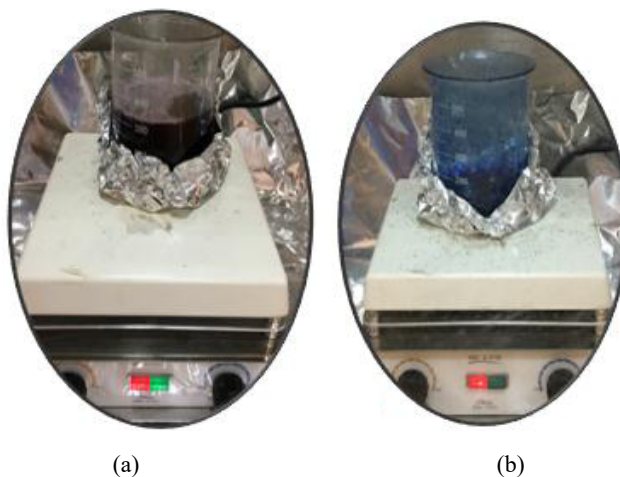


Figure 2 Photographic image of the combustion reaction (a) before and (b) after combustion (see online version for colours)



The combination was heated for burning to combustion at 400°C on hot plate along five hours two then dried via using furnace at 400°C along solely 2 hour of time. Firstly, the combustion reaction is the drying (evaporation) from water and then converted into

gases, and it used to be found raises in the viscosity of the mixture, and hastily, the torch happen the waist of melting-pot. Figure 2 confirmed photos before the reaction vessel and after material as-synthesised were once accumulated in powder-like form and subjected to characterisation and in addition processing.

2.2 Characterisation methods

Thermal evaluation used to be finished the usage of thermal analyser (STA-1000 model) from LINES, Germany with rate heating 10°C per min under neath surrounding environment. The type of the structural property and homogeneity degree of the product used to be tested by JEOL (JDX-8030) XRD analysis the usage of Cu-K α radiation. To analyses the form and morphology structure of the synthesised LiNiCoO₂, SEM snap shots were taken by way of JEOL (JSM-840A). The FTIR for powder have been investigated with range (350–7,800) cm^{-1} by triglycine sulphate (TGS) detectors, S.C. detector mercury cadmium tolerate (HgCdTe) MCT and Alpha from Bruker Optics made in Germany.

3 Results and discussion

The weight loss in the temperature range $25\text{--}100^{\circ}\text{C}$ is attributed to the weight loss of water molecules that are held through nanoparticle. Organic aspects belonging to (CO₂, H₂, N₂) two team of lithium nitrate and cobalt nitrate decomposed up till $\sim 512.7^{\circ}\text{C}$, after which, no more weight loss is view. Clearly, a low weight absence happened between ~ 383.4 and 512.7°C .

Table 1 FTIR for LCNO powder were investigated by $350\text{--}7,800\text{cm}^{-1}$

<i>Gaind bond value (cm⁻¹)</i>	<i>Our search</i>
< 400	Stretching vibration of LiO ₆
545–585	Bending and stretching vibration of M-O, O-M-O (M = Li, Ni and Co)
1,000–1,500	Stretching vibration of H-O-H bond
1,377.22	Stretch vibration C-C
2,843.17	Expansibility vibration, NH ₂ = 3,400–4,000 stretch vibration, O-H of glycerol and water = 3,600–4,000 stretch vibration and C-H = 2,800–3,200 stretch vibration

The infrared bonds of solids are ordinarily allotted to shaking of particles within the precious stone grid. FTIR of LCNO immaculate stage was recorded in Table 1 and examined tests is appeared in Figures 3(a) and 3(b). FTIR spectra of the nano-compound powders gotten some time recently after combustion response. Lithium powder gotten by damp chemical precursory union, show a vibration between 900 and 500 cm^{-1} that demonstrate the presence of O-M-O vibrations. Within the spectra from Figure 3(a), it can be watched a really tactful broadband from 500 to 1,000 cm^{-1} that coincident with Tarascon (2010) is related to the O-M-O vibration. Over 1,000 cm^{-1} , a few crests too were watched that likely are characteristic of a few H-O-H vibration starting from hydration of the tests some time recently the investigation, this assertion with Markova (2010).

Figure 3 Results of the thermo-gravimetric analysis (TGA) measurements (see online version for colours)

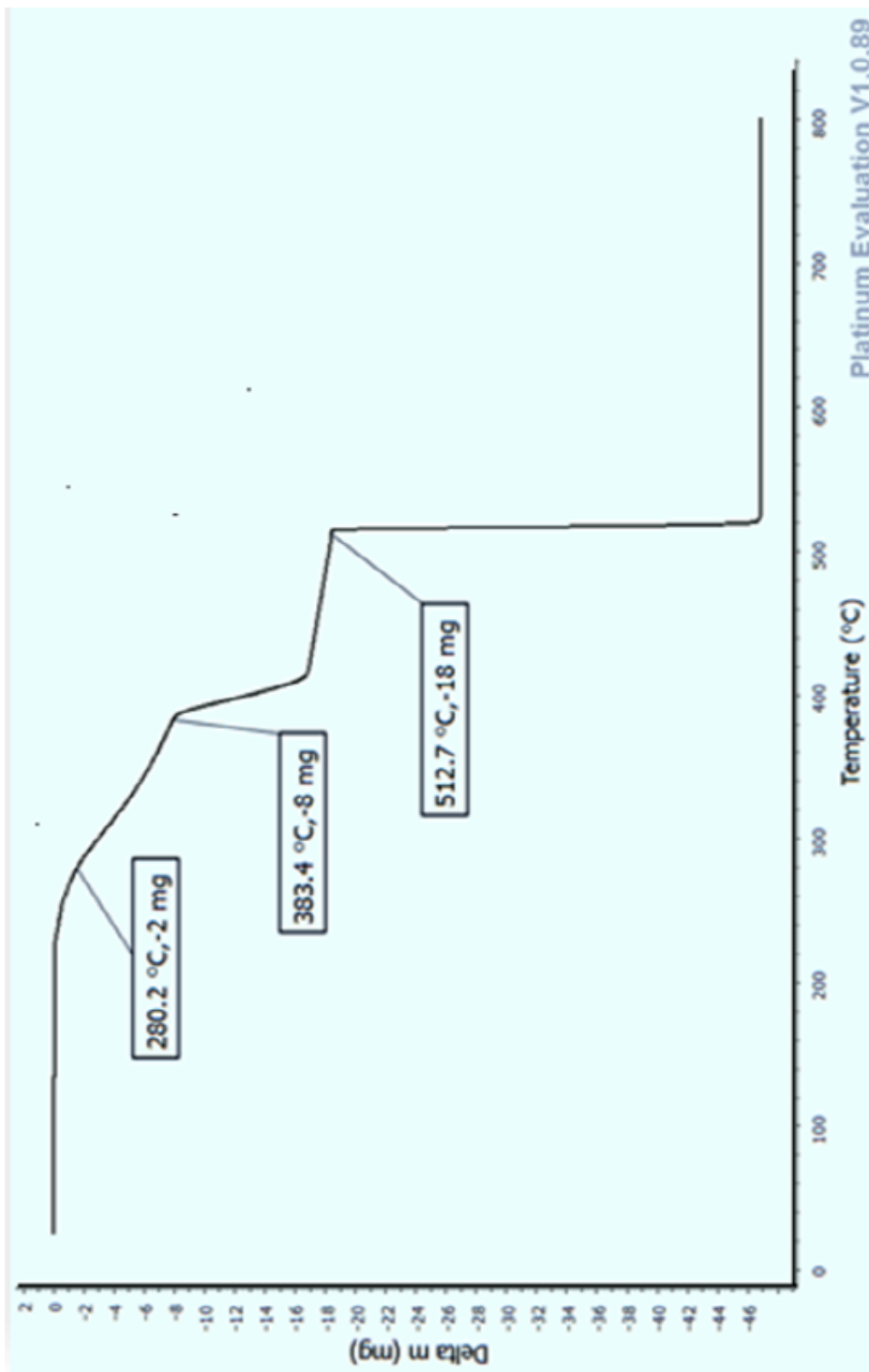
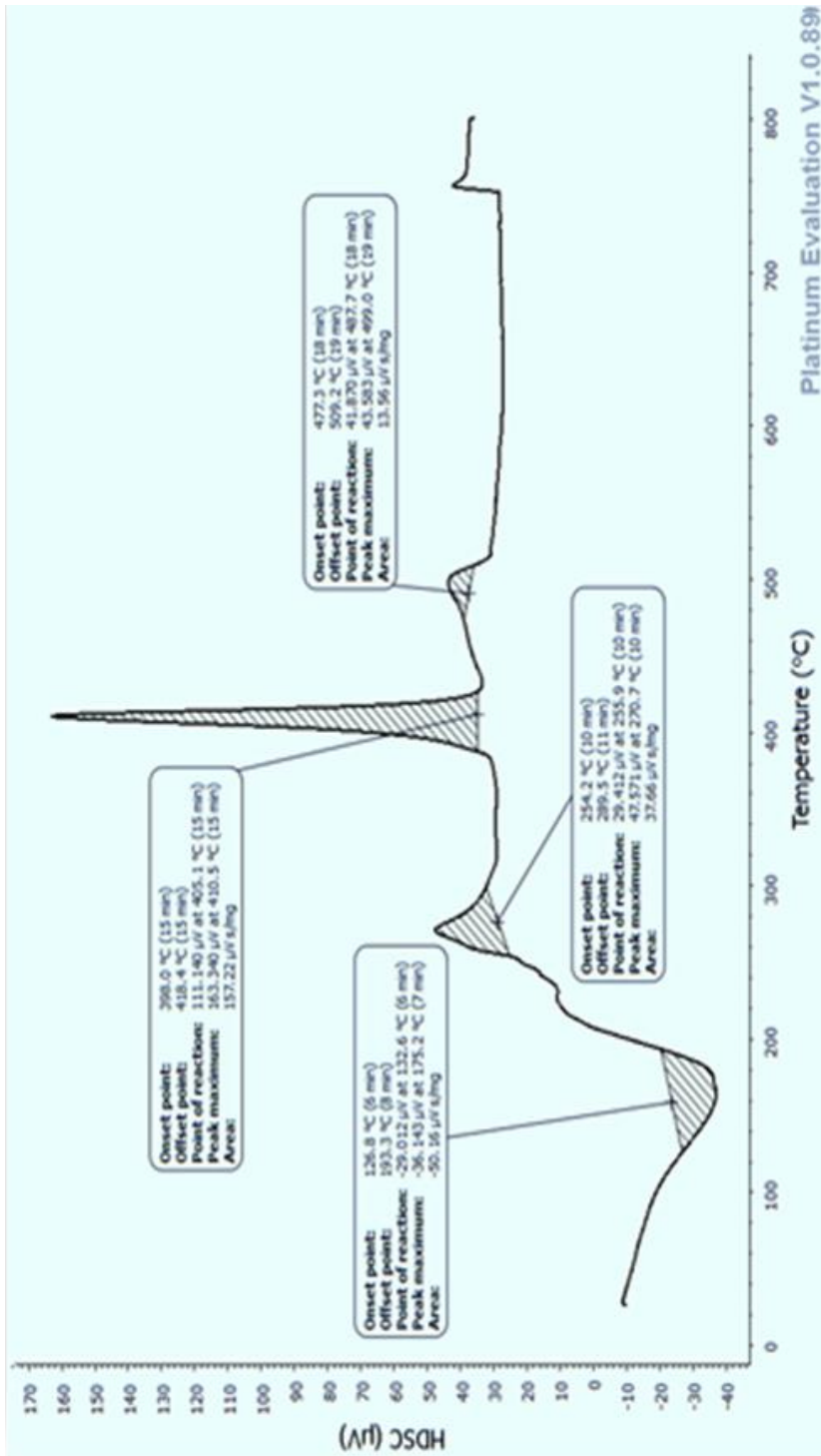
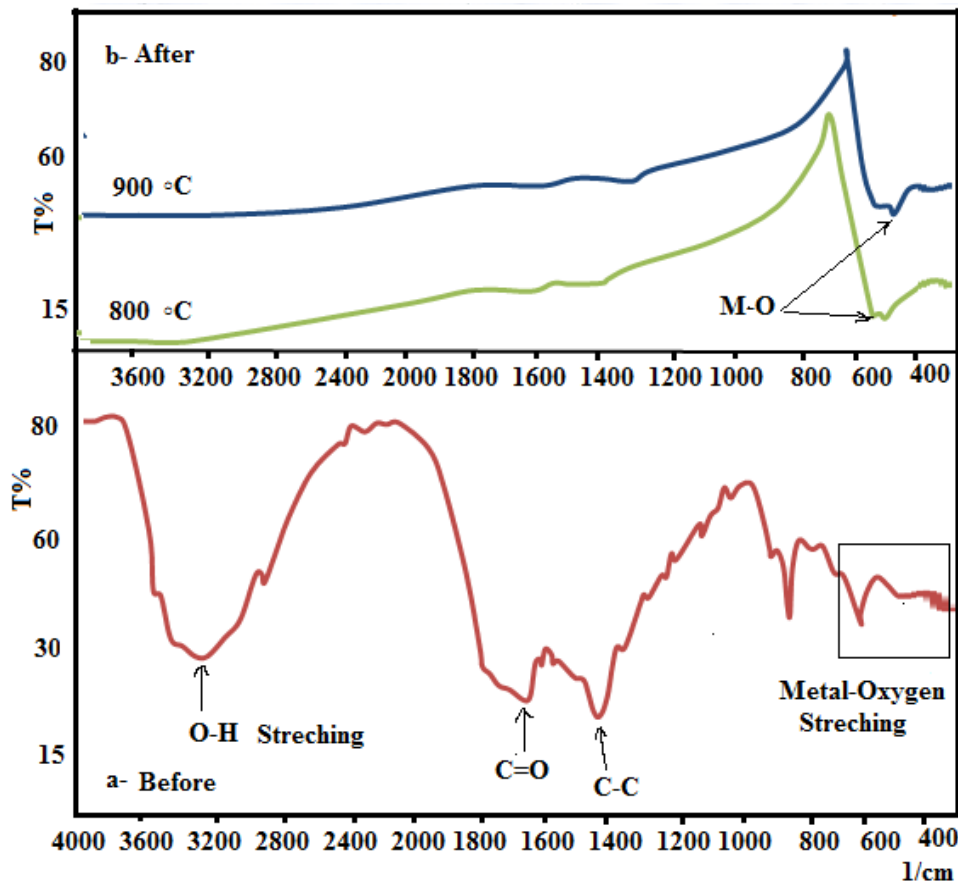


Figure 3 Results of the thermo-gravimetric analysis (TGA) measurements (see online version for colours)



Also, as can be seen from Figure 4(a), before annealing, there are multiple and clear peaks that appear from 500 to 1,000, which indicate the presence of oxides appear at the peaks 547 and 585 are the present metal – oxygen and at the top (1,377.22) a clear presence of the urea located in the middle of the interaction C-C.

Figure 4 FTIR spectra of the LCNO arranged by implies of wet-chemistry procedures and unmistakable temperature (a) before and (b) after annealing (see online version for colours)



At the top 1,600.97, there is a peak resulting from the overlapping of multiple summers of the current, which extends to 172.13 C = O and C-H at the top 2,843.17. A clear presence of the present within the complex glycerol.

The region stretching from 3,271 and 3,431, respectively, which seemed to overlap, representing the amine and hydroxyl groups of urea and glycerol, respectively, as the region indicate 3,200–4,000 to the presence of moisture partially.

From Figure 4(b), after annealing at the temperature of 800°C, we notice very clearly and sharply of the oxide peak, this indicating the presence of metals oxide and the absence of the amine and hydroxyl groups of urea and glycerol. In addition to evaporation of all moisture aggregates that were visible before the annealing.

Also, there are multiple and overlapping peaks in the region between 400 and 500 indicating that there are a number of oxides with oxygen-metal bonds, and that a clear and broad peak exists. Another proof of the presence of a mixture of oxides in the model in peak 3,429 is the assertion that there is no water. This is a proof that the model was burned well.

The XRD designs of the polycrystalline powders as-prepared and tempering in discuss at before and after annealing at 800°C and 8 h are appeared within Figure 5. The powders synthesised with urea, and glycerol have diffraction crests that can be ordered well with the reference of layer structure LiCoNiO_2 (ICDD 056-1441) which could be a hexagonal precious stone framework with space bunch R-3m(166). Subsequently, the temperatures of combustion between the fills and metal nitrates were ideal for the arrangement of the immaculate stage. In any case, the tests are tempering at 400°C and 2h from Figure 5(a) and after annealing at 800°C from Figure 5(b) due to nearness of natural compounds because it was appeared within the warm investigation. Table 2 appears the normal crystallite sizes of the powders as-prepared and strengthening, which were decided from the complete width at half most extreme (FWHM) of the most strongly crests (003), (101), (006), (012), (104), (015), (107), (018), (110) and (113) using Scherer equation (Zheng et al., 2011).

Figure 5 Illustrated X-ray analysis of LiCoNiO_2 got by glycerol, urea-nitrate (see online version for colours)

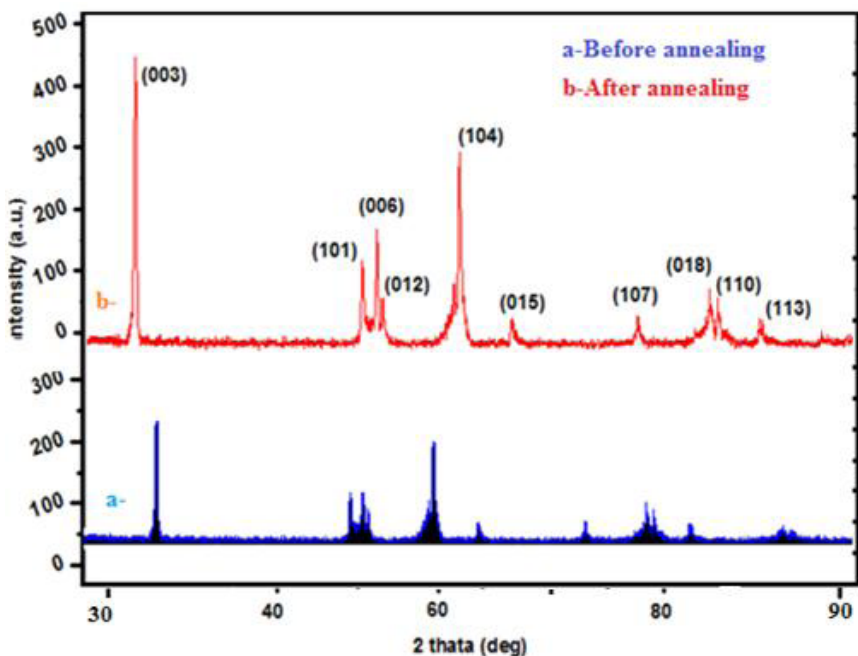


Table 2 Illustrated the XRD parameters for synthesised LiCoNiO₂ by combustion reaction at heat treatment

<i>2</i> Theta (deg)	<i>FWHM</i> (deg)	<i>d</i> (Å)	<i>I</i> / <i>I</i> ₁	<i>Miller indices</i>	<i>Grain size</i> (Å)
38.38	0.187	2.343	86	(003)	7.85
44.33	0.151	2.041	5	(101)	9.98
44.60	0.181	2.029	100	(006)	8.27
44.78	0.111	2.022	16	(012)	13.50
64.90	0.213	1.435	9	(104)	7.71

It can be seen that the tempering of the powders at 800°C and 4 h increment the sizes of the crystallites. XRD estimations illustrated NiOH was artificial if the cobalt was attached. Widened nickel hydroxide will lead to more dynamic destinations uncovered the glycerol, conceivably, first reason for direct urea fuel cell (DUFC) advancement. Arrange to assist clarify seen why the expansion of Co would impact catalytic movement. The X-ray diffraction designs show that the tests containing urea brought about within the arrangement of crystalline powders and the nearness of hematite as an auxiliary stage. The tests containing glycerol displayed as it were the arrangement of crystalline and monophases LiCoNiO₂. The normal crystallite measure was 18 and 65 nm.

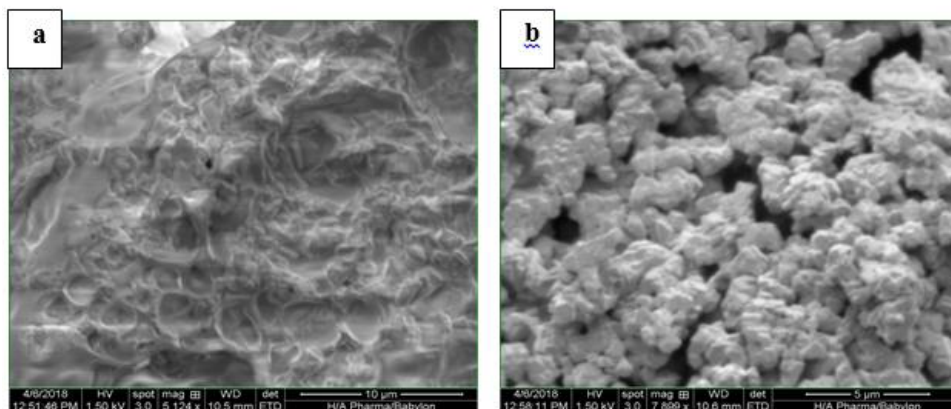
Figure 6 SEM micrographs for LiCoNiO₂ obtained by urea and glycerol nitrate for (a) before annealing and (b) after annealing at 800°C

Figure 6 presents SEM micrographs the LiCoNiO₂ gotten by arrangement combustion with the powers glycerol and urea, which uncover agglomerated, permeable, and sponge-like morphologies of the particles, these characteristics are detailed within the logical writing for the arrangement combustion synthesis (Xu et al., 2015; Haider et al., 2016), this is often due to advancement of the huge sum of vaporous items amid combustion, which produces highly porous voluminous powders, and these parameters

subordinate of fuel utilised within the amalgamation. The glycerol appears higher permeable than urea and citric corrosive, be that as it may, it is vital to note that the tests appears agglomeration of nanoparticles, but the urea appears non-metric gems of octahedral shape of measure greater than the glycerol and citric corrosive, which is reliable with the values calculate from XRD patterns by Scherrer condition. Hence, these comes about indicate that an alter within the fuel utilised within the union lead to noteworthy changes within the microstructure of the powders. When glycerol is utilised, the porosity is higher that urea and citric corrosive, because it was appeared within the SEM micrographs, for that reason, the scrambling of light increments. In this manner, the increase in diffuse reflectance of glycerol is ascribed to extend of scrambling light by tall porosity of the powder, due to produced gasses amid the combustion response, though within the case of urea and citric corrosive the gasses are lower than glycerol. Moreover, the molecule measure is lower within the glycerol, which increments the surface range, and subsequently the diffusing of light too increases (Patil et al., 2011; Jameel et al., 2016).

4 Conclusions

X-ray direction designs appeared arrangement of this composite materials and grid parameter (a, b) were measured. SEM investigation confirmed the arrangement less than one micro molecule nature of LiNiCoO₂ nanoparticles.

This combustion strategy may well a promising strategy to synthesising LiCoNiO₂. Exploratory conditions will fine-tuned to obtain diminished particles and further electrochemical studies might be carried out to setup finest cathode fabric for LIB applications.

References

- Al-Douri, Y., Haider, A.J., Reshak, A.H., Bouhemadou, A. and Ameri, M. (2012) 'Structural investigations through cobalt effect on ZnO nanostructures', *Optik – International Journal for Light and Electron Optics*, Vol. 127, No. 20, pp.10102–10107.
- Christian, J., Alain, M., Ashok, V. and Karim, Z. (2016) *Lithium Batteries Science and Technology*, pp.29–68, Springer, Cham, ISBN: 978-3-319-19108-9.
- Feng, C., Li, H., Zhang, C., Guo, Z., Wu, H. and Tang, J. (2012) Synthesis and electrochemical properties of nonstoichiometric Li-Mn-spinel (Li_{1.02} Mx Mn_{1.95} O_{4-y} Fy) for lithium ion battery application', *Electrochimica Acta*, Vol. 61, pp.87–93.
- Haider, A.J., AL-Rsool, R.A. and Abdullah, A.Q. (2018a) 'Electrical properties and electrochemical impedance spectroscopy for LiCo_{0.5}Ni_{0.45}Ag_{0.05}O₂ powder', *International Conference on Advances in Sustainable Engineering and Applications (ICASEA)*, Wasit University, Kut, Iraq.
- Haider, A.J., AL-Rsool, R.A. and Haider, M.J. (2018b) 'Morphological and structural properties cathode compound material for lithium-ion battery', *Plasmonics*, Vol. 13, No. 5, pp.1649–1657.
- Haider, A.J., Al-Tabbakh, A.A., Al-Zubaidi, A.B. and Rsool, R.A. (2018c) 'Preparation and characterization of LiCo_{0.5}Ni_{0.45}Ag_{0.05}O₂ cathode material for lithium-ion battery', *Journal of Materials Science: Materials in Electronics*, <https://doi.org/10.1007/s10854-018-9451-z>.
- Haider, A.J., Thamir, A.D., Najim, A.A. and Ali, G.A. (2016) 'Improving efficiency of TiO₂: Ag/Si solar cell prepared by pulsed laser deposition', *Plasmonics*, Vol. 12, No. 1, pp.105–115.

- Jameel, Z.N., Haider, A.J., Taha, S.Y., Gangopadhyay, S. and Bok, S. (2016) 'Evaluation of hybrid sol-gel incorporated with nanoparticles as nano paint', *AIP Conference Proceedings*, Vol. 1758, No. 1.
- Markova, D. (2010) 'Infrared spectroscopy investigation of metallic nanoparticles based on copper, cobalt, and nickel synthesized through borohydrate reduction method', *Journal of the University of Chemical Technology and Metallurgy*, Vol. 45, No. 4, p.351.
- Patil, V.P., Pawar, S., Chougule, M., Godse, P., Sakhare, R., Sen, S. and Joshi, P. (2011) 'Effect of annealing on structural, morphological, electrical and optical studies of nickel oxide thin films', *Journal of Surface Engineered Materials and Advanced Technology*, Vol. 1, No. 2, pp.35–41.
- Tarascon, J.M. (2010) 'Key challenges in future Li-battery research', *Philosophical Transactions of the Royal Society of London A: Mathematical, Physical and Engineering Sciences*, Vol. 368, No. 1923, pp.3227–3241.
- Xu, W., Zhang, H., Li, G. and Wua, Z. (2015) 'Nickel-cobalt bimetallic anode catalysts for direct urea fuel cell', *PMC*, Vol. 68, pp.859–870.
- Zheng, J.M., Wu, X.B. and Yang, Y. (2011) 'A comparison of preparation method on the electrochemical performance of cathode material $\text{Li}[\text{Li}_{0.2}\text{Mn}_{0.54}\text{Ni}_{0.13}\text{Co}_{0.13}]\text{O}_2$ for lithium ion battery', *Electrochimica Acta*, Vol. 56, No. 8, pp.3071–3078.