Preparation of MgO–MnO₂ nanocomposite particles for cholesterol sensors

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ABSTRACT

Various nanoparticles have been developed for bio-applications. However, nanocomposites particles, (NCPs) with effective and ability to sensing cholesterol, are still needed. Herein, we present new cholesterol and pH-responsive NCPs as sensor particles to promote sensitivity. The traditional method of mixing and grinding was used to fabricate the oxides of magnesium and manganese MgO–MnO₂ NCPs with different mixing ratios. Structural properties, detection of cholesterol, and pH sensitivity were examined. Results proped the high efficiency of MgO–MnO₂ NCPs compared with individual oxides (MgO and MnO₂), low response time, while the analytical results confirmed the homogeneous structure of MgO–MnO₂ NCPs. Particle size distribution results for NCPs were within 16.4 to 100 nm, which makes it promising in medical and bio-applications.

1 Introduction

Intelligent use of physiological activity monitoring gadgets such as heart rate monitors, step or calorie counting devices, or even lactate threshold monitoring patches became senior. The aim is to get information without exceeding especially patient privacy and for monitored healthcare and patients [1]. With the emergence of the coronavirus disease-2019 (COVID-19), the importance of monitoring the cardiovascular and vital functions has increased for workers and patients [2].

All of this increased the demand for bio- and biomedical sensors which are exceptional electronic devices that can transduce biomedical signals into

easily measurable electric signals [3]: physical, chemical, and biological quantity sensors. Biological quantity sensors are sensors used to detect biological parameters such as tissues, cells, enzymes, antigens, antibodies, receptors, hormones, cholic acid, acetylcholine, cholesterol, serotonin, DNA, and RNA, and other proteins. Cholesterol is an organic molecule that is created by the human body to maintain cell membrane temperature. High levels of cholesterol can narrow the arteries and raised the risk of heart disease. Cholesterol has several types such as low-density lipoprotein (LDL) or bad cholesterol, high-density lipoprotein (HDL), or good cholesterol—the bad cholesterol eater and Lp (a) cholesterol. Metal oxides MO_x such as SnO₂, ZnO, CuO, CeO, NiO, and MnO₂ have different sensing

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ranges to Cholesterol [4, 5]. Another biomarker sign is monitoring the stability of pH of organs such as the heart diverse MOx for pH sensors, IrO_2 , WO_3 , RuO_2 , TiO_2 , Ta_2O_5 , and ZnO which were employed. [6–8] Magnesium oxide MgO is a wide inorganic substance including a broad band-gap. It has been used in various fields such as in bio-medicine [9, 10]. MgO has been applied for bone repair, the release of heartburn, and in the sensitive stomach, pharmaceutical applications. Recently, MgO nanoparticles have been used in cancer therapy besides having considerable activities as an antibacterial agent [11]. MgO nanoparticles [12, 13] Several researchers present various approaches for MgO nanostructure synthesis like nano-bundles (thermal evaporation method) [14], nanorods (magnesium chloride solution was bio-reduced to magnesium oxide nanorods using *Tectona grandis* leaves extract) [15, 16]; nanosheets cryptocrystalline magnesite was converted into MgO nanosheets using thermal activation and ball milling [17]. Nano-flower displayed a biological method for synthesis of magnesium oxide (MgO) nano-flowers by using Aqueous Rosemary [18]. Nanobelt was obtained by magnesium nitrate decomposition using the direct arc plasma jet chemical vapor deposition technique, with a molybdenum substrate [19]. Nanotrapods (Tubular tetrapod magnesium oxide (tt- MgO) can be synthesized by thermal evaporation of Mg metal powder with a pre-grown tetrapod ZnO template.) [20] Hierarchical nanostructure (also 3D hierarchical flower-like MgO microstructure (HFI- MgO), which was synthesized by chemical precipitation method [21, 22]. Hollow mesoporous MgO nanospheres (HMMN) have been demonstrated by the coating of carbon spheres template with MgO nanoparticles, then the calcination in the presence of air to remove the carbon core at elevated temperature [23].

On the one hand, the synthesis of the MgO nanocomposite gets received widespread attention, like CuO-MgO nanocomposites (prepared by a solvothermal procedure for catalytic applications) [24, 25], $\text{Y}_2\text{O}_3\text{-MgO}$ nanocomposite (by ceramics powder sintering) [26], CdO-MgO (microwave-assisted method) [27–29], $\text{MgO-Fe}_2\text{O}_3$ nanocomposite (was prepared via a simple precipitation route) [30, 31], ZnO-MgO (formed by the sol-gel method) [29, 30], $\text{Ag}_2\text{S-MgO/graphene}$ (was prepared via sol-gel/ultrasound method) [32, 33] $\text{Al}_2\text{O}_3\text{-MgO}$ (synthesized by a sol-gel process) [34], $\text{CaCO}_3\text{-MgO}$ (synthesized MgO nanoparticle-coated eggshell particles (defined as CaCO_3/MgO nanocomposites) and

subsequently manufactured a biomimetic active scaffold via the chemical crosslinking) [35]. $\text{Y}_2\text{O}_3\text{-MgO}$ (colloidal technique and SPS spark-plasma-sintering (SPS) techniques.) [36].

Yet the mill method is still an exciting technique to prepared nanomaterials [37, 38] and nanocomposite such as WC-MgO [39]. The ball milling method with High-energy was a functional procedure fit for achieving solid-state of various alloy systems, nanocrystalline and nanocomposite materials. The main characterized of the milling method was industrial significance, room temperature process and high-thermal stability. The important parameters were milling time to milling speed (TV) and ball-to-powder weight ratio (BPR) [40]. Manganese oxide MnO_2 is also interesting due to its properties and dependable in sensors by their different nanostructure like tunnel structures [41], nanosheet [42], nanowires [43], nanorods [44], nano-flower [45], nanosphere [46]. Different methods were used to analyze cholesterol percentages; electrochemical method is a simple and promising method procedure for biosensor applications. The pH sensors depend on diverse mechanisms (e.g. potentiometric, conductometric, chemi-resistors, and ion-sensitive field-effect transistor (ISFET), etc.) [47, 48].

Our work issues a wide insight into the MgO-MnO_2 nanocomposite particles utilizing different weight mixture percentages to construct highly sensitive and rapidly responsive cholesterol sensors.

2 Experimental methods

2.1 Preparing MgO-MnO_2 nanocomposite

The experimental steps to prepare MgO/MnO_2 nanocomposite particles NCPs are listed below.

The oxides MgO: MnO_2 (high purity 99% provided from HUBEI AOKS BIO-TECH CO., LTD.) with (10 μm) average size. The mix weight ratios were five (100:0, 75:25, 50:50, 25:75, 0:100 wt %) denominate by $\text{NCP}_{\text{S1-5}}$, respectively. After determining the exact weights and ratios of the raw materials, we added them to ethanol Alcohol suspension and homogenized using the ultrasonic device about 30 min type (Hielscher) with the power of 2 W and frequency 26 kHz and 1000 rpm to ensure uniformity of distribution. Then, the mixes were milled by using a friction mill for 4 h.

After that drying process within an oven of type IMS was used at a temperature of $60 \pm 5^\circ\text{C}$, for 3 h.

The gotten mixed powders were sintered at 400 °C for 1 h by MTI CORPORATION, GSL1600X furnace.

2.2 Inspection of MgO/MnO₂ nanocomposite

2.2.1 Structural properties

Energy-dispersive x-ray (EDX) spectra were recorded using a scanning electron microscope (SEM) study, carried out by electron gun tungsten, secondary electrons detector mode, ultrahigh vacuum (10^{-6} bar), 20 kV of accelerating voltage, and variable working distance; SEM MAG was 10.0KX (Type TESCAN VEGA III) (CZECH). The nature and the crystal growth of the MgO/MnO₂ nanocomposites were examined by X-ray diffraction XRD. The measurements were made by using Philips PW 1050 X-ray diffractometer of (1.5406) Å; copper K α radiation was used. The particle size test for particles was performed by (Brookhaven Nano-Brook 90 plus USA).

2.2.2 Samples preparation

The method was used to determine the cholesterol in blood samples. The samples were prepared by using 5 ml of blood sample centrifuged (by Centrifuge type 903485 Taiwan) to get 1 ml of serum. The separated part was extracted and mixed using 10 mL of total dissolved solids water TDS water and sonicated for 15 min with (Biobase Biodustry (Shandong) Co., Ltd., China).

2.2.3 Cholesterol measurement

The dispersed NCPs have higher sensitivity, functionalized electrodes and conductivity. Figure 1

shows the experimental steps for measurement. Sensor preparation for cholesterol measurements was performed by adding 500 μ L (Syringe 1750N CTC, (26/51/AS)) at 5 wt% NCPs into an Eppendorf tube (Conical Tubes). A 40 μ L of the solution was drop cast onto SPCE for cyclic amperometry measurements using Drop Send DRP-STAT400 (Electrochemical measurements using two electrodes to measure current nA range). Then, a volume of 40 μ L of cholesterol solutions, with various concentrations (25, 50, 75, 100, 125, and 150) μ L, was added into another Eppendorf tube, mixed, and kept for 20 min. After that, it is compared with the readings of the tests of real patients of different ages and different blood groups to verify the values obtained. The readings were using the Radox Rx Daytona.

2.2.4 pH measurements

Effect of Ionic Strength. To check the effect of ionic strength on the pH sensing ability of NCPs, 50 μ L fraction of NCPs₁₋₅ solution and a volume of 50 μ L of cholesterol solutions of different concentrations were taken. The pH measurements were done by using pH meter type CRISON.

3 Results and discussion

3.1 X-ray diffraction results

Figure 2 shows the XRD patterns of specimens with angular scanning range of (30 – 80°), for the MgO pure and modified with MnO₂ nanocomposites at a: (0, 25, 50, 75, 100 wt %) in (a, b, c, d, and e) represented by NCPs₁₋₅.

Fig. 1 Schematic experimental steps for measurement, **a** mixing, **b** dropping and **c**measuring

The NCPs1 Fig. 2a is MgO polycrystalline nanoparticles consisted of pure single-phase cubic system agreed with the JCPDS database card No. (30–0794). The XRD pattern of the MgO shows five peaks at 38.44°, 44.37°, 59.18°, 64.52°, and 73.33° attributed to the (222), (400), (511), (440) and the (620) planes; the (400) is the preferred orientation formed. No crystalline impurity phases were observed [49, 50].

On the one hand, the NCPs5 Fig. 2e is β -MnO₂ polycrystalline nanoparticles consisted of pure single tetragonal phase system agreed with the JCPDS database card No. (24-0735).

The XRD pattern of the MnO₂ also shows six peaks at 37.33°, 42.83°, 56.65°, 59.37°, 72.26°, and 72.38° attributed to the (101), (111), (211), (220), (301), and the (112) planes; the (101) and (211) are the preferred orientation planes formed [51].

The samples NCPs₂, NCPs₃, and NCPs_{2, 3, 4} in Fig. 1b–d, respectively, for MgO–MnO₂ nanocomposites, both pure phases MgO and β -MnO₂ were presented in different planes intensities, consistent with the proportions of mixing and preparation. And to verify the homogeneity of the formed nanocomposites particles additional realization was carryout via calculation of the lattice constants, lattice strain, and texture coefficient.

Lattice strain δ percentage broadening is caused by changes in displacements of the atoms concerning their reference lattice position, evaluated from equation [52–54]:

Fig. 2 XRD patterns of **a** NCPs₁ and **b** NCPs₂, **c** NCPs₃, **d** NCPs₄, **e** NCPs₅

$$\delta = \frac{D_s - D_m}{D_s} \times 100\% \quad (1)$$

where D_s and D_m are the standards and the measured lattice constants. For a certain crystal plane (hkl) in a polycrystalline particle, the orientation preferred was represented by [55]:

$$T_C = \left(\frac{I_m/I_o}{M^{-1} \sum \frac{I_m}{I_o}} \right) \quad (2)$$

where T_C is the texture coefficient for a certain (hkl) plane, I_m is the measured intensity value, I_o is the JCPDS standard intensity value of the corresponding powder and M is the number of reflections observed in the XRD trace. The structural result from XRD data of MgO, MnO₂ NPs, and NCPs is in Table 1. The lattice constants were very close to card standard values, while the lattice strain and the texture coefficient can be acquired under the present synthetic conditions varying between pure NPs and NCPs, by the difference in lattice constants and the mixing ratio of the composites particles.

3.2 SEM results

Figure 3 illustrates the SEM images and particle size distribution of **a**: MgO NPs and **b**: NCPs₂, **c**: NCPs₃, **d**: NCPs₄, **e**: MnO₂ NPs₅. In Fig. 3a the MgO NPs have a homogeneous distribution of a rod-like composition and Avg. Diameter about 156.91 nm and cumulative lengths up to 10 μ m, agree with [50], while Fig. 3e shows the SEM images and particle size distribution of MnO₂ NPs₅. It shows a very fine spherical structure with Avg. Diameter: 102.71 nm; the aggregation takes place to beat the higher surface energy because of the higher surface to volume ratio [56]. On the other hand, in Fig. 3b for mixing ratio

Table 1 Structural result from XRD data of mix MgO, MnO₂ NPs, and NCPs

		a Å	c Å	δ (*10 ⁻³)	T_c
NPs	NCPs ₁	8.118	—	0.05	0.9
NCPs	NCPs ₂	8.117	—	0.3	0.88
		4.399	2.872		
	NCPs ₃	8.117	—	0.7	0.65
		4.399	2.873		
	NCPs ₄	8.117	—	0.2	0.8
		4.399	2.873		
NPs	NCPs ₅	4.398	2.874	0.02	0.92

Fig. 3 SEM images and particle size of **a** NPs and **b** NCPs₂, **c** NCPs₃, **d** NCPs₄, **e** NPs₅

75%:25%, the rod-like composition, and Avg. Diameter present in Fig. 3. For MgO NPs reduced with present MnO₂ and formation MgO–MnO₂ nanocomposite with higher density and smaller Avg. The diameter reaches 123.53 nm. And with an increase in the percentage of manganese oxide to 50% the Avg. Diameter continues to drop to 106.71 nm. And the composition turns into a semi-hollow (or tunnel) structure form composed of equal proportions of the oxides. This unique hollow shape transforms with an increasing proportion of MnO₂ to a smooth semi-spherical shape compared to the previous composition of the ratios, Avg. Diameter return rising to 119.95 nm. This diversity in morphology is due to the mechanism of energy transfer during the processes of grinding and mixing. In Fig. 3d the NCPs₄ has a semi-spherical structure.

3.3 EDX results

To identify the element distribution, EDX analysis of the specimens was performed. The EDX analysis shown in Fig. 4 the Micrograph image for MgO, MnO₂ nanoparticles (in a and e), and MgO–MnO₂ nanocomposites (in b, c, and d). Figure 4 shows the presence of Mg and O associated with the main percentage of the constituent elements (parent composition). The same goes for Fig. 4e, Mn, and O elements. Elemental analysis of MgO: MnO₂ mixed composite particles was confirmed by EDX characterization. The mixes M2, M3, and M4 contained Mg, Mn, and O elements [57, 58].

3.4 Particle size results

Figure 5 demonstrates the particle-size distribution on the MgO and MnO₂ NPs (a: NCPs, e: NCPs₅) with NCPs at different ratios (b: NCPs₂, c: NCPs₃, d: NCPs₄).

In Fig. 5a the PZ of MgO was 1138 nm and 65% of the formed particles were below 1138 nm. In Fig. 5e the PZ of MnO₂ was 364 nm and 65% of the formed particles were below this value.

Composite nanoparticles were in b: 100 nm, in c: 83 nm, and in d: 16.4 nm. It should be noted that this study showed that 50–65% of the formed particles were in the nano-range which can have a significant effect on the performance of the sensors.

Fig. 4 EDX analysis ratio of **a** NPs and **b** NCPs₂, **c** NCPs₃, **d** NCPs₄, **e** NPs₅

3.5 Cholesterol sensor results

Cholesterol sensor results are presented in Fig. 6; the current peak obtained from different Cholesterol concentrations is plotted as a function of sensing time. It shows a high rate increase during the first 20 s, followed by a slight increase up to 80 s of examination time, and the result tends to be stable in the last 10 s of the examination time. This behavior is observed in all the prepared mixtures of nanocomposite oxides. The minimum detection time was 10 s in Fig. 6b: NCPs₂ and in the c: NCPs₃. And that is due to the hollow structure that increases the surface area of the reaction for this sensor. Through this analysis and by comparison with the measured values for actual patients, a match was found, suggesting the benefit of rapid detection compared to conventional methods.

3.6 pH-sensing

Figure 7 shows the results of pH for the prepared nanocomposites particles. In this test the molecular formula is C₂₇H₄₆O and heavy molecular weight

(386.7 g/mol), topological polar surface Area is 20.1 Å² with a density of about 1.067 g/cm³, a white, crystalline substance that is odorless and tasteless prepared cholesterol was synthesized in vitro. Moreover, several healthy men and patients, male and female, were examined and compared with the laboratory value.

The results showed that all the prepared nanocomposites particles were highly sensitive to the change in the pH values under examination conditions. The results of healthy people subjects ranged from 7.35 to 7.45 for males and females, and for all nanoparticles superposed. The preeminent nanocomposites particles mixed were NCPs₃ which matched with the reference sample. This is due to the hollow structure and the higher specific surface area presented in SEM in Fig. 3c. In un-healthy people's subjects, the detection of pH was more effective. As it recorded different heights and was in NCPs₂ and NCPs₃ better compared with single oxides (NCPs₁

and NCPs₅), as the measurement sensitivity reached 2% for both men and women [59, 60].

Moreover, RX Daytona plus by Randox Laboratories Ltd, UK device was used to determine the real values of patients. As shown in Table 1 the gender, average age, nanocomposites particles sensitivity *S*%, test results *t_R*, and a number of patients No. were illustrated.

Table 2 shows that all manufactured sensors have different detection ratios and match the actual results. And the best rate (in case of the absence of high cholesterol) was 77.3% for 23 males and 17 females with average ages 45, 48 years, respectively, for NCPs₃. In patients with high cholesterol, they are classified into two types: the first type Positive but Low, the sensors showed a remarkable match with the actual values. The best of them were 85% of males, number 11 with an average age and 53 years, and 84.1% for a female with number 34 an average age was 48 years. The second type is Positive High,

Fig. 5 Particle size distribution patterns of **a** NCPs and **b** NCPs₂, **c** NCPs₃, **d** NCPs₄, **e** NCPs₅

Fig. 6 Current results of
a MgO NPs and **b** NCPs₂,
c NCPs₃, **d** NCPs₄, **e** MnO₂
 NPs₅

the correspondence rate with the actual results increased to 93%. With the actual results, in the case of males, number 45, with an average age of 63 years, and in the case of females, 92%, number 14, with an average age of 55 years.

Moreover, the rest of the detectors also achieved a match with the actual values and there remains more than one option available, but NCPs₃ an ideal choice for pH sensing.

Fig. 7 The pH- results of **a** NCPs₁ and **b** NCPs₂, **c** NCPs₃,
d NCPs₄, **e** NCPs₅

Table 2 Sensitivity results for examined patients

Gender	No	Avg.Age	t_R mg/dl	S%					Result
				NCPs ₁	NCPs ₂	NCPs ₃	NCPs ₄	NCPs ₅	
Male	23	45	173	11.3	22.8	77.3	18.7	5.8	Negative
Female	17	38	155	11.3	22.8	77.3	18.8	5.8	Negative
Male	11	53	275	41.3	51.6	85.1	41.9	23.2	Positive/ Low
Female	34	48	242	41.6	52.9	84.1	43.8	23.2	Positive/ Low
Male	45	63	368	67.6	77.6	93.2	57.7	37.3	Positive/High
Female	14	55	338	68	77.3	92.2	57.3	39.1	Positive/High
Ref	1	–	100	50					Normal

4 Conclusion

In this work, we successfully synthesized different nanocomposites particles from MgO–MnO₂ with varied crystal structures via a milling method for cholesterol sensor applications. By altering the synthesis mixing ratio, three shapes were obtained: rods, hollows, and semi-spherical nanocomposites particles. The equivalent mixing ratio produces hollow tunnel structures that were systematically synthesized. Every combination has a different response to cholesterol. PH-responsive and uncharged nanocomposite particles were able to detect the change in the pH in a time of up to 10 s and with an efficiency exceeding 50%. Moreover, the recorded current values were stable after only 80 s, allowing accurate values to be obtained.

References

1. S. Dinc, B. Şahin, T. Kaya, *Mater. Sci. Semicond. Process.* **105**, 104698 (2020)
2. E. Driggin, M.V. Madhavan, B. Bickdeli, T. Chuich, J. Laracy, G. Biondi-Zoccai, T.S. Brown, C.D. Nigoghossian, D.A. Zidar, J. Haythe, D. Brodie, J.A. Beckman, A.J. Kirtane, G.W. Stone, H.M. Krumholz, S.A. Parikh, *J. Am. Coll. Cardiol.* **75**(18), 2352–2371 (2020)
3. D. Feng, *Biomedical Information Technology*, 2nd edn. Biomedical Engineering (2020) pp. 51–79, eBook ISBN: 9780128160350
4. B. Sahin, T. Kaya, *Mater. Res. Express* **6**(4), 042003 (2019)
5. M.T. Awayiz, E.T. Salim, *Mater. Sci. Forum* **1002**, 200–210 (2020)
6. A.D. Faisal, R.A. Ismail, W.K. Khalef, E.T. Salim, *Opt. Quant. Electron.* **52**, 1–12 (2020)
7. L. Manjakkal, D. Szwagierczak, R. Dahiya, *Prog. Mater. Sci.* **109**, 100635 (2020)
8. E.T. Salim, A.I. Hassan, S.A. Naaes, *Mater. Res. Express* **6**(8), 086416 (2019)
9. C.W. Wong, Y.S. Chan, J. Jeevanandam, K. Pal, M. Bechelany, M. Abd Elkodous, G.S. El-Sayyad, *J. Clust. Sci.* **31**, 367–389 (2020)
10. M.T. Awayiz, E.T. Salim, *AIP Conf. Proc.* **2213**(1), 020247 (2020)
11. J. Jeevanandama, Y.S. Chana, M.K. Danquaha, *Prot. Met. Phys. Chem. Surf.* **55**(2), 288–301 (2019)
12. A.A. Abdullah-Hamead, *Mater. Sci. Forum* **1002**, 319–330 (2020)
13. E.T. Salim, R.A. Ismail, H.T. Halbos, *Mater. Res. Express* **6**(11), 116429 (2019)
14. Q. Yang, J. Sha, L. Wang, Y. Wang, X. Ma, J. Wang, D. Yang, *Nanotechnology* **15**(8), 1004 (2004)
15. H.A. Maishera, F.A. Kuta, J.O. Tijani, N.U. Adabara, A.S. Adedeji, J.D. Bala, *J. Bio-Sci.* **27**, 109–120 (2019)
16. M.A. Fakhri, M.J. AbdulRazzaq, A.A. Alwahib, W.H. Muttlak, *Opt. Mater.* **109**, 110363 (2020)
17. V. Masindi, *Mater. Today* **38**(2), 1077–1087 (2021)
18. Y. Abdallah, S.O. Ogunyemi, A. Abdelazez, M. Zhang, X. Hong, E. Ibrahim, A. Hossain, H. Fouad, B. Li, J. Chen, *Hindawi Biomed Res. Int.*, **2019**, Article ID 5620989, 8 (2019)
19. H. Li, M. Li, W. Guo, X. Wang, C. Ge, B. Yang, *Electrochim. Acta* **123**, 103–110 (2014)
20. J. Zhao, L. Qin, Y. Hao, Q. Guo, F. Mu, Z. Yan, *Microchim. Acta* **178**, 439–445 (2012)
21. P. Karacabey, S. Döven, D. Uzunoğlu, A. Özer, *Arab. J. Sci. Eng.* **44**, 9951–9964 (2019)
22. E.T. Salim, R.A. Ismail, H.T. Halbos, *Appl. Phys. A* **126**, 891 (2020)
23. M. Zare, *J. Dispersion Sci. Technol.* (2020). <https://doi.org/10.1080/01932691.2020.1737103>
24. S.K. Alla, A.D. Verma, V. Kumar, R.K. Mandal, I. Sinha, N.K. Prasad, *RSC Adv.* **66**, 61927–61933 (2016)

25. M.A. Fakhri, E.T. Salim, M.H.A. Wahid, U. Hashim, Z.T. Salim, *J. Mater. Sci.: Mater. Electron.* **29**(11), 9200–9208 (2018)
26. H.J. Ma, W.K. Jung, Y. Park, D.K. Kim, *J. Mater. Chem. C* **41**, 11096–11103 (2018)
27. Z.T. Salim, U. Hashim, M.K.M. Arshad, M.A. Fakhri, E.T. Salim, *Mater. Res. Bull.* **86**, 215–219 (2017)
28. K. Kannan, D. Sivasubramanian, P. Seetharaman, S. Sivaperumal, *Optik* **204**, 164221 (2020)
29. M.A. Fakhri, U. Hashim, E.T. Salim, Z.T. Salim, *J. Mater. Sci. Mater. Electron.* **27**(12), 13105–13112 (2016)
30. A. Azhari, M. Sharif, S.F. Golestanifard, A. Saberi, *Mater. Chem. Phys.* **124**(1), 658–663 (2010)
31. F.G. Khalid, A.S. Ibraheam, M.A. Fakhri, N.H. Numan, *AIP Conf. Proc.* **2213**(1), 020204 (2020)
32. K. Tamizh Selvi, K. Alamelu Mangai, M. Priya, S. Sagadevan, *Physica B* **594**, 412355 (2020)
33. E.T. Salim, R.A. Ismail, M.A. Fakhri, B.G. Rasheed, Z.T. Salim, *Iran. Sci. Technol. Trans. A* **43**(3), 1337–1343 (2019)
34. H. Wang, G. Li, A. Fakhri, *J. Photochem. Photobiol. B* **207**, 111882 (2020)
35. H.A. Sallal, A.A. Abdul-Hamead, F.M. Othman, *Eng. Technol. J.* **38 Part A**, 586–593 (2020)
36. Y. Huang, Y. Ji, Z. Kang, F. Li, S. Ge, D. Yang, J. Ruan, X. Fan, *Chem. Eng. J.* **395**(1), 125098 (2020)
37. L. Liu, K. Morita, T.S. Suzuki, B. Kim, *Ceram. Int.* **46**(9), 13669–13676 (2020)
38. R.A. Ismail, E.T. Salim, W.K. Hamoudi, *Mater. Sci. Eng. C* **33**(1), 47–52 (2013)
39. Q.A. Hamad, M.S. Abed, *J. Mech. Eng. Res. Dev. JMERC* **42**(5), 153–157 (2019)
40. J. Ma, S.G. Zhu, C.X. Wu, M.L. Zhang, *Mater. Des.* **30**(8), 2867–2874 (2009)
41. Y. Dai, J. Huang, H. Zhang, C. Chiun Liu, *Sens. Actuators B* **281**, 746–750 (2019)
42. Q. Zuo, Y. Chen, Z. Chen, R. Yu, *Talanta* **209**, 120528 (2020)
43. Q. He, J. Liu, X. Liu, G. Li, D. Chen, P. Deng, J. Liang, *Electrochim. Acta* **296**, 683–692 (2019)
44. T. Chhabra, A. Kumar, A. Bahuguna, V. Krishnan, *Vacuum* **160**, 333–346 (2019)
45. L. Hao, L. Xue, F. Huang, G. Cai, W. Qi, M. Zhang, Q. Han, Z. Wang, J. Lin, *Micromachines* **11**(3), 281 (2020)
46. T. Li, Z. Wang, D. Jiang, H. Wang, W. Lai, Y. Lv, Y. Zhai, *Sens. Actuators B* **290**, 535–543 (2019)
47. N. Salehifar, Z. Zarghami, M. Ramezani, *Mater. Lett.* **167**, 226–229 (2016)
48. M.A. Fakhri, Y. Al-Douri, U. Hashim, E.T. Salim, D. Prakash, K.D. Verma, *Appl. Phys. B* **121**(1), 107–116 (2015)
49. X. Wang, Y. Li, *J. Am. Chem. Soc.* **124**(12), 2880–2881 (2002)
50. A.L. Abed, W.K. Khalef, E.T. Salim, *J. Phys. Conf. Ser.* **795**(1), 012031 (2021)
51. A.A. Abdul-Hamead, *AIP Conf. Proc.* **1968**, 030017 (2018)
52. M.U. Khalid, M.F. Warsi, I. Shakir, M.F.A. Aboud, M. Shahid, S.S. Shar, S. Zulfikar, *Ceram. Int.* **46**(7), 9913–9923 (2020)
53. E.T. Salim, *Surf. Rev. Lett.* **20**(05), 1350046 (2013)
54. M.A.M. Hassan, M.F.H. Al-Kadhemy, E.T. Salem, *Int. J. Nanoelectron. Mater.* **8**(2), 69–82 (2014)
55. A. Kadhim, E.T. Salim, S.M. Fayadh, A.A. Al-Amiery, A.H. Kadhum, A.B. Mohamad, *Sci. World J.* **2014**, Article ID 490951, 6 (2014)
56. X. Liu, S. Fu, C. Huang, *Powder Technol.* **154**, 120–124 (2005)
57. M. Rezaei, M. Khajenoori, B. Nematollahi, *Powder Technol.* **205**, 112–116 (2011)
58. N.M. El-Sawy, A.I. Raafat, N.A. Badawy, A.M. Mohamed, *Int. J. Biol. Macromol.* **142**, 254–264 (2020)
59. Z. Zhang, Y. Ji, W. Chen, *Chin. J. Chem. Eng.* **28**, 1405–1414 (2020)
60. E.T. Salim, J.A. Saimon, M.K. Abood, M.A. Fakhri, *Opt. Quant. Electron.* **52**, 4631–4716 (2020)

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