

Full Paper

Synthesis of Pt-SWCNTs Conductive Nanocomposite by Microwave Heated Polyol Strategy; Application for Amplification of 5-Fluorouracil Anticancer Drug Electrochemical Sensor

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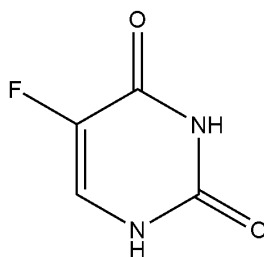
Abstract- In this study, a simple strategy was described for the synthesis of Pt-SWCNTs conductive nanocomposite by microwave heated polyol method and nanocomposite characterized by EDS, FESEM, and XRD method. The Pt nanoparticles were decorated at the surface of SWCNTs with a diameter of 22.3 nm. The synthesized nanocomposite was used for modification of the carbon paste electrode (CPE) in the presence of n-hexyl-3-methylimidazolium hexafluorophosphate (nH3MHP) and paraffin oil as binders. The Pt-SWCNTs/nH3MHP/CPE was showed a good catalytic effect for electro-oxidation of the 5-fluorouracil anticancer drug in aqueous solution. In comparison to CPE, the Pt-SWCNTs/nH3MHP/CPE increased oxidation current of 5-fluorouracil (~4.47 times) and reduce oxidation potential of this anticancer drug ~125 mV. On the other hand, Pt-SWCNTs/nH3MHP/CPE was successfully used for the determination of 5-fluorouracil anticancer drugs in injection samples with acceptable recovery data (96.13%-103.5%). According to recorded results, the sensor has a powerful tool for determination of 5-fluorouracil anticancer drug in real samples.

Keywords- 5-Fluorouracil; Anticancer drug; Microwave heated polyol method; Pt-SWCNTs conductive nanocomposite

1. INTRODUCTION

Attention to new nanostructures for use in various industries has grown significantly in recent years [1-10]. Meanwhile, conductive nanomaterials have received more attention than other nanostructures due to their widespread use in various applications such as fuel cells, batteries, electrical chips, and electrochemical sensors [11-15]. According to scientific reports, carbon nanotubes and metal compounds, especially platinum nanoparticles, have very high electrical conductivity and have played a decisive role in industrial applications [16-20]. For example, platinum nanoparticles are well used in the preparation of fuel cells [21], and carbon nanotubes are one of the most widely used nanomaterials in the preparation of electron exchange sensors [22-30]. Based on this, it can be predicted that nanocomposites based on a combination of carbon nanotubes and platinum nanoparticles can provide high electrical conductivity for the design of electrochemical sensors [31].

5-Fluorouracil (Aduvicol) (Scheme 1) is anticancer that is prescribed to treat pancreatic cancer, colon cancer, cervical cancer, breast cancer, esophageal cancer, and stomach cancer over the past 20 years [32]. Due to the high side effects of this drug in the chemotherapy process, dose control used in the treatment of patients is very important and necessary [33].



Scheme 1. Structure of 5-Fluorouracil

Therefore, fast and sensitive analysis of 5-fluorouracil in a biological sample that is a major problem in the chemotherapy process [33-35]. Many analytical sensors were used for the determination of 5-fluorouracil in biological samples [36-38]. In between, electrochemical methods showed more advantages due to fast response and portable ability [39-47]. However, high over-voltage and weak redox signals of 5-fluorouracil are the most important problems in the design of electrochemical sensors for electro-analysis of 5-fluorouracil [48]. Therefore, using conductive mediators for the fabrication of a 5-fluorouracil electroanalytical sensor is very important. In between, attention to ionic liquids and carbon-metal based nanocomposite could help to create highly sensitive electro-analytical sensors [49-55]. Nanomaterials with incredible properties have been used as widely used materials in many chemical techniques [56-64]. High conductivity is main advantages of nanomaterials for using in electrochemical sensors [65]. Therefore, the present study described fabrication of Pt-SWCNTs/nH3MHP/CPE as a highly sensitive electrochemical sensor for determination of 5-

fluorouracil. Two-fold amplification of CPE with Pt-SWCNTs nanocomposite and nH3MHP created a highly sensitive condition for trace level analysis of 5-fluorouracil and improved ability of sensor for determination of this anticancer drug in real samples.

2. EXPERIMENTAL

2.1. Materials and instrument

5-Fluorouracil $\geq 99\%$, chloroplatinic acid hydrate $\geq 99.9\%$, n-hexyl-3-methylimidazolium hexafluorophosphate, SWCNTs were purchased from Sigma-Aldrich. Sodium hydroxide, graphite powder 99.9%, ammonia anhydrous $\geq 99.98\%$, and paraffin oil were purchased from Across Company. A μ -Autolab PGSTAT was used for electrochemical investigation using Ag/AgCl/KCl sat as a reference electrode. X'Pert Pro and Mira-3-XMU instruments were used for XRD and FESEM investigations.

2.2. Synthesis of Pt-SWCNTs/nH3MHP/CPE

The ethylene glycol solutions and H_2PtCl_6 were used as precursors for the synthesis of Pt/SWCNTs by microwave heating strategy. The 50 mL ethylene glycol + 0.8 mL potassium hydroxide (0.4 M) was mixed in an erlenmeyer flask under stirring for 10 min and 2.0 mL H_2PtCl_6 solution (0.05 M) was added into erlenmeyer flask and stirring was continued for 10 min. In the next step, 0.1 g of SWCNTs was added into the erlenmeyer flask and dispersed using an ultrasonic strategy for 30 min in distilled water. The erlenmeyer flask was placed in the center of a household microwave oven and heated for 3 min under microwave power of 600 W. The obtained powder was dried at 120 °C for 12 h. 2.3. Preparation of Pt-SWCNTs/nH3MHP/CPE

The Pt-SWCNTs/nH3MHP/CPE was prepared by mixing 920 mg graphite powder + 80 mg Pt-SWCNTs in the presence of 10 cc ethanol as a solvent into mortar and pestle. After evaporation of ethanol, paraffin + nH3MHP (80:20 v:v) was added as a binder and the resulting paste was added end of the glass tube.

3. RESULTS AND DISCUSSION

3.1. Characterization of Pt-SWCNTs nanocomposite

The Pt-SWCNTs were characterized by XRD, EDS, and FESEM methods. The XRD pattern of Pt-SWCNTs showed five planes with miller indexes [002] relative to the carbon nanotubes phase and [111]; [200]; [220] and [311] relative to Pt nanoparticles with JCPDS Card 04-0802 (Fig. 1). The diameter of Pt nanoparticle was calculated 22.3 nm using the Debye-Scherrer equation.

FESEM images of Pt-SWCNTs nanocomposite clearly showed the presence of single-wall carbon nanotubes decorated by Pt nanoparticles (Fig. 2A). According to the FESEM image, Pt

nanoparticle is a presence at the surface of SWCNTs. On the other hand, EDS analysis data show the presence of C, Pt elements that confirms the purity of synthesized Pt-SWCNTs nanocomposite (Fig. 2B).

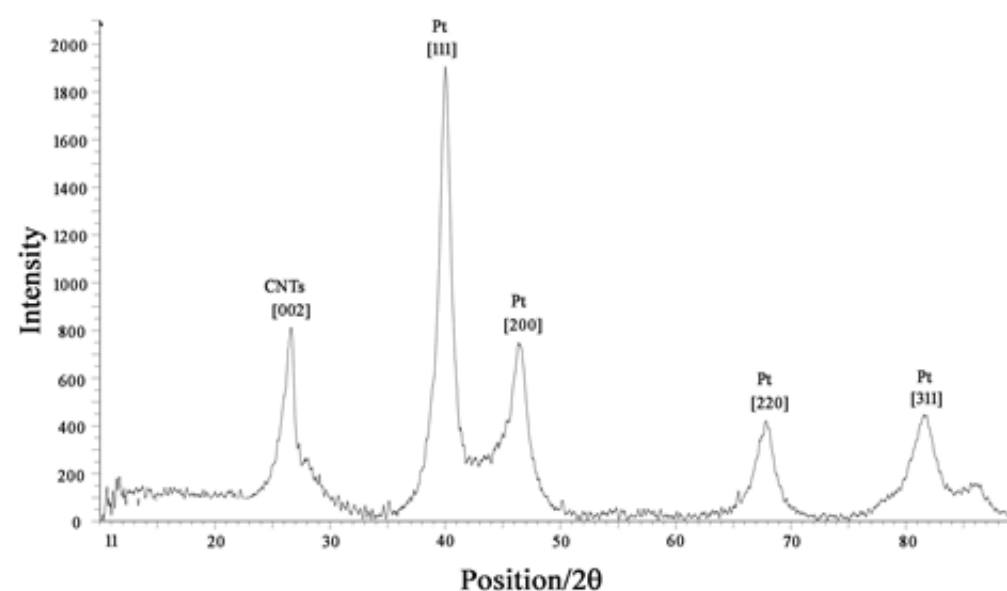


Fig. 1. XRD pattern of Pt-SWCNTs nanocomposite

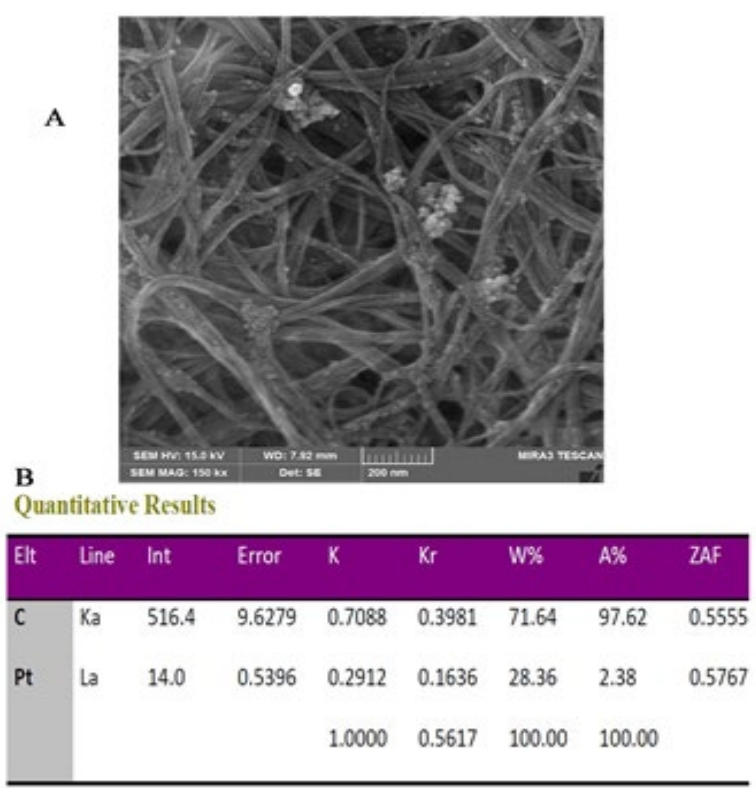


Fig. 2. A) FESEM image and B) EDS analysis data of Pt-SWCNTs nanocomposite

3.2. Electrochemical investigation

Due to the uracil structure and reported papers, the oxidation behavior of 5-fluorouracil is dependent on pH changes [36]. Therefore, the factor of pH was optimized in the first step. Fig. 3 inset showed a square wave voltammograms of 80 μM 5-fluorouracil in the pH range of 7.5-9.5. As can be seen in fig. 3, with moving pH=7.5 to pH=9.5 the oxidation potential of 5-fluorouracil shifted to a negative value with equation $E = -0.0588 \text{ pH} + 1.3012$ ($R^2 = 0.9982$) that confirm the value of electron and H^+ in redox mechanism of this anticancer drug is equal. On the other hand, maximum oxidation current for 5-fluorouracil was detected at pH=8.5 and this condition was selected for the next steps.

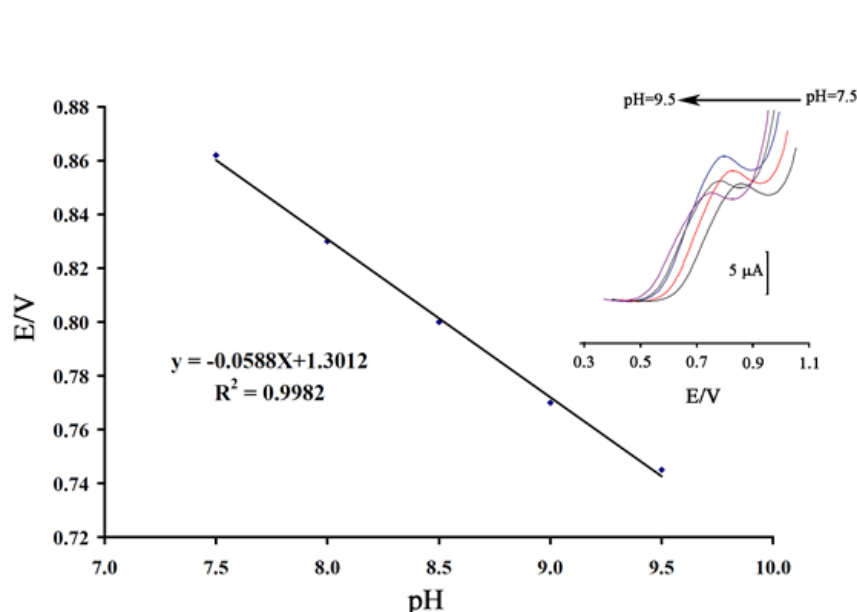


Fig. 3. E-pH curve for electro-oxidation of 80 μM 5-fluorouracil at a surface of Pt-SWCNTs/nH3MHP/CPE. Inset) Relative Square wave voltammograms for electro-oxidation of 80 μM 5-fluorouracil

For study role Pt-SWCNTs and nH3MHP on modification of CPE at the surface of Pt-SWCNTs/nH3MHP/CPE, square wave voltammograms of 80 μM 5-fluorouracil was recorded at the surface of CPE (fig. 4 curve a), Pt-SWCNTs/CPE (Fig. 4 curve b), nH3MHP/CPE (Fig. 4 curve c) and Pt-SWCNTs/nH3MHP/CPE (Fig. 4 curved).

Oxidation currents 3.78 μA , 9.44 μA , 13.5 μA , and 16.9 μA were recorded for curves a-d, respectively. As can be seen, with the modification of CPE with Pt-SWCNTs or nH3MHP, the oxidation signal of 5-fluorouracil was improved. In addition, the best oxidation signal was recorded at the surface of Pt-SWCNTs/nH3MHP/CPE that confirm the modification of CPE with Pt-SWCNTs and nH3MHP could be improved oxidation current of 5-fluorouracil ~ 4.47 times. Fig. 4 inset shows current density data relative to recorded square wave voltammograms

of 80 μM 5-fluorouracil at the surface of different electrodes that confirm good electrical conductivity of Pt-SWCNTs and nH3MHP as mediators.

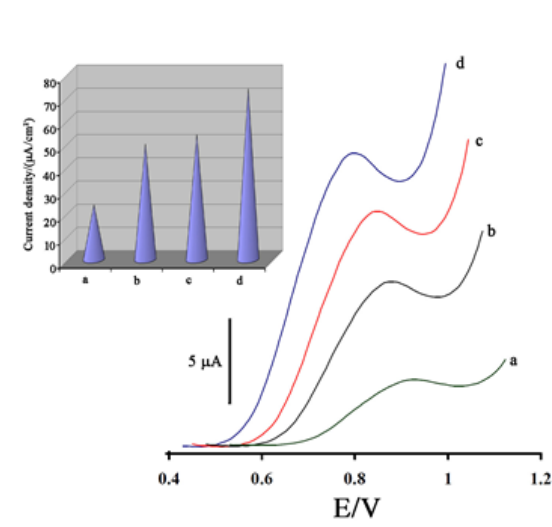


Fig. 4. SWVs of 80 μM 5-fluorouracil at surface of a) CPE, b) Pt-SWCNTs/CPE, c) nH3MHP/CPE and d) Pt-SWCNTs/nH3MHP/CPE. Inset) relative Square wave voltammograms recorded at the surface of different electrodes.

The CVs of 700 μM 5-fluorouracil at scan rate ranges 50-200 mV/s was recorded at surface of sensor (Fig. 5 inset).

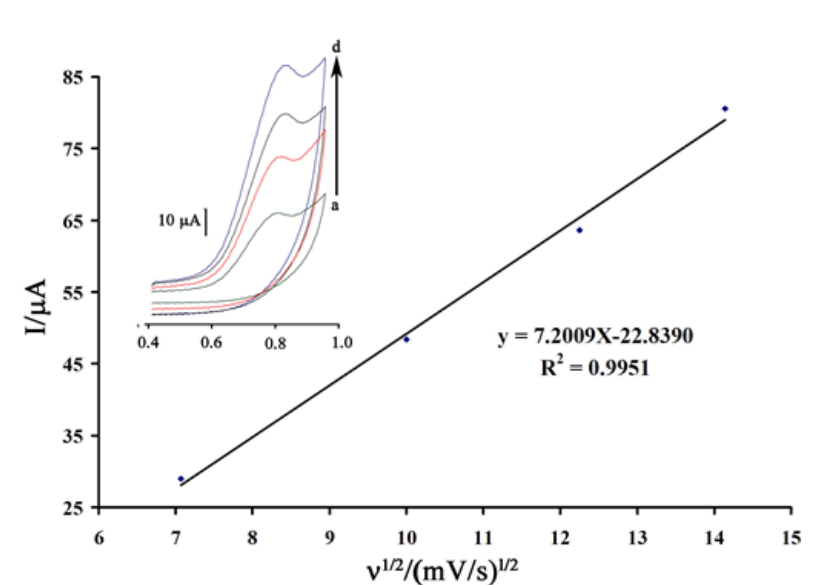


Fig. 5. Current- $v^{1/2}$ curve for electro-oxidation of 700 μM 5-fluorouracil at surface Pt-SWCNTs/nH3MHP/CPE. Inset) CVs of 700 μM 5-fluorouracil recorded at scan rates a) 50, b) 100, c) 150 and d) 200 mV/s.

A linear relation between oxidation current of 5-fluorouracil and $v^{1/2}$ with the equation of $I = 7.2009 v^{1/2} - 22.8390$ ($R^2 = 9951$) was observed that confirm diffusion process [66-70] for the electro-oxidation reaction of 5-fluorouracil at surface of sensor (Fig. 5).

SWV method was used for study linear dynamic range and limit of detection 5-fluorouracil at the surface of Pt-SWCNTs/nH3MHP/CPE and results showed a linear relation between current and 5-fluorouracil concentration in the range 1.0 nM-520 μ M (Fig. 6). The detection limits of 5-fluorouracil were calculated 0.4 nM using 3Sb/m equation.

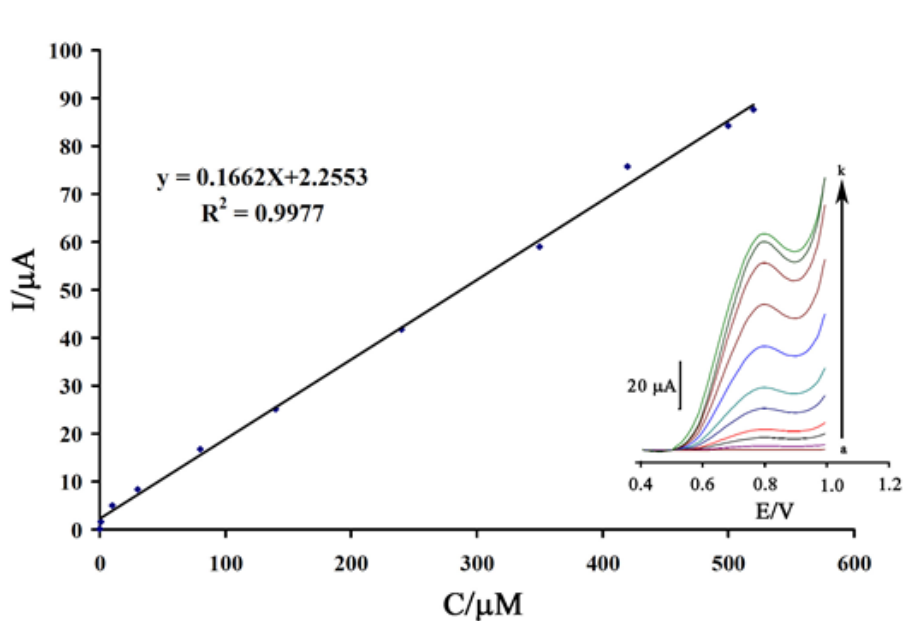


Fig. 6. Current-concentration diagram for electro-oxidation of 5-fluorouracil at surface Pt-SWCNTs/nH3MHP/CPE. Inset) Square wave voltammograms a) 0.001; b) 1.0; c) 10; d) 30; e) 80; f) 140; g) 240; h) 350; i) 420; j) 500 and k) 520 μ M recorded at surface of Pt-SWCNTs/nH3MHP/CPE.

The stability of Pt-SWCNTs/nH3MHP/CPE as a new electrochemical sensor was investigated for a determination of 10.0 μ M 5-fluorouracil in period time 30 days. According to recording results, a 90% initial oxidation signal of 5-fluorouracil remains after 30 days that confirm the good stability of Pt-SWCNTs/nH3MHP/CPE as a new electrochemical sensor for determination of 5-fluorouracil.

The selectivity of Pt-SWCNTs/nH3MHP/CPE as a new electrochemical sensor for determination of 10 μ M 5-fluorouracil was check in the presence of some organic and inorganic interference with acceptable error 5%. Results showed nor 1000-fold of Na^+ , Cl^- , K^+ , and Br^- , 750-fold of vitamin B2, and glucose and 300-fold ascorbic acid did not affect the selectivity.

Table 1. Real sample analysis data for determination of 5-fluorouracil using Pt-SWCNTs/nH3MHP/CPE

Sample	Added (μM)	Expected (μM)	Founded (μM)	Recovery%
Injection	---	---	2.51 ± 0.21	---
	5.00	7.51	7.22 ± 0.46	96.13
Pharmaceutical serum (dextrose saline)	---	---	<LOD	---
	10.00	10.00	10.35 ± 0.76	103.5

The ability of Pt-SWCNTs/nH3MHP/CPE as a new electroanalytical sensor was a check for the determination of 5-fluorouracil in injection and pharmaceutical samples without any pre-preparation. The real sample analysis data are a presence in table 1 and confirm the high-performance ability of Pt-SWCNTs/nH3MHP/CPE for the determination of 5-fluorouracil in real samples.

4. CONCLUSION

In this research, a highly sensitive voltammetric sensor was fabricated for the determination of 5-fluorouracil. For this goal, CPE was amplified with Pt-SWCNTs and nH3MHP. The Pt-SWCNTs/nH3MHP/CPE was showed catalytic activity for the determination of 5-fluorouracil and increased oxidation current of 5-fluorouracil ~ 4.47 times. In addition, the Pt-SWCNTs/nH3MHP/CPE was effectively used for the determination of 5-fluorouracil in injection samples.

REFERENCES

- [1] Y. Orooji, M. Ghanbari, O. Amiri, and M. Salavati-Niasari, *J. Hazard. Mater.* 389 (2020) 122079.
- [2] S. Malekmohammadi, H. Hadadzadeh, S. Rezakhani, and Z. Amirghofran, *ACS Biomater. Sci. Eng.* 5 (2019) 4405.
- [3] A. Amiri, M. Baghayeri, and S. Nori, *J. Chromatogr. A* 1415 (2020) 20.
- [4] H. Karimi-Maleh, M. Shafieizadeh, M. A. Taher, F. Opoku, E. M. Kiarai, P. P. Govender, S. Ranjbari, M. Rezapour, and Y. Orooji, *J. Mol. Liq.* 298 (2020) 112040.
- [5] S. Malekmohammadi, H. Hadadzadeh, and Z. Amirghofran, *J. Mol. Liq.* 265 (2018) 797.
- [6] M. Ghasemi, A. Khataee, P. Gholami, R. D. C. Soltani, A. Hassani, and Y. Orooji, *J. Environ. Manage.* 267 (2020) 110629.

- [7] S. Rayati, and S. Malekmohammadi. *J. Exp. Nanosci.* 11 (2016) 872.
- [8] P. Mehdizadeh, Y. Orooji, O. Amiri, M. Salavati-Niasari, and H. Moayedi, *J. Clean. Product.* 252 (2020) 119765.
- [9] S. Malekmohammadi, H. Hadadzadeh, H. Farrokhpour, and Z. Amirghofran, *Soft matter* 14 (2018) 2400.
- [10] H. Karimi-Maleh, B. G. Kumar, S. Rajendran, J. Qin, S. Vadivel, D. Durgalakshmi, F. Gracia, M. Soto-Moscoso, Y. Orooji, and F. Karimi, *J. Mol. Liq.* 314 (2020) 113588.
- [11] V. Arabali, S. Malekmohammadi, and F. Karimi, *Microchem. J.* (2020) 105179.
- [12] A. L. Sanati, and F. Faridbod, *Int. J. Electrochem. Sci.* 12 (2017) 7997.
- [12] M. A. Khalilzadeh, A. Hojjati-Najafabad, M. S. Rahmanpour, F. Karimi, H. Zabihi-Feyzaba, S. Malekmohammadi, S. Agarwal, and V. K. Gupta, *Int. J. Electrochem. Sci.* 15 (2020) 6969.
- [13] H. Karimi-Maleh, F. Karimi, S. Malekmohammadi, N. Zakariae, R. Esmaili, S. Rostamnia, M. L. Yola, N. Atar, S. Movagharneshad, S. Rajendran, A. Razmjou, Y. Orooji, S. Agarwal, and V. K. Gupta, *J. Mol. Liq.* 310 (2020) 113185.
- [14] F. Faridbod, and A. L. Sanati, *Curr. Anal. Chem.* 15 (2019) 103.
- [15] H. Karimi-Maleh, F. Karimi, M. Alizadeh, and A. L. Sanati, *Chem. Rec.* 20 (2020) <https://doi.org/10.1002/tcr.201900092>
- [16] A. L. Sanati, F. Faridbod, and M. R. Ganjali, *J. Mol. Liq.* 241 (2017) 316.
- [17] H. Karimi-Maleh, K. Cellat, K. Arikan, A. Savk, F. Karimi, and F. Şen, *Mater. Chem. Phys.* 250 (2020) 123042.
- [18] H. Karimi-Maleh, and O. A. Arotiba, *J. Colloid Interface Sci.*, 560 (2020) 208.
- [19] M. Fouladgar, *J. Electrochem. Soc.* 165 (2018) B559.
- [20] A. Alkhalilzadeh, *Anal. Bioanal. Electrochem.* 12 (2020) 780.
- [21] F. Hasché, M. Oezaslan, and P. Strasser, *Phys. Chem. Chem. Phys.* 12 (2010) 15251.
- [22] A. A. Ensafi, H. Karimi-Maleh, and S. Mallakpour, *Colloids Surf. B.* 104 (2013) 186.
- [23] A. A. Ensafi, H. Karimi-Maleh, and S. Mallakpour, *Electroanalysis* 23 (2011) 1478.
- [24] M. A. Khalilzadeh, H. Karimi-Maleh, A. Amiri, and F. Gholami, *Chin. Chem. Lett.* 21 (2010) 1467.
- [25] H. Karimi-Maleh, F. Tahernejad-Javazmi, V. K. Gupta, H. Ahmar, and M. H. Asadi, *J. Mol. Liq.* 196 (2014) 258.
- [26] J. B. Raof, R. Ojani, H. Karimi-Maleh, M. R. Hajmohamadi, and P. Biparva, *Anal. Methods* 3 (2011) 2637.
- [28] S. S. Moshirian-Farahi, H. A. Zamani, and M. Abedi, *Eurasian Chem. Commun.* 2 (2020) 702.
- [29] H. Pyman, H. Roshanfekar, and S. Ansari, *Eurasian Chem. Commun.* 2 (2020) 213.
- [30] P. Prasad, P. Aruna, K. Prabhakar, and N.Y. Sreedhar, *Chem. Methodol.* 2 (2018) 277.
- [31] L. Hosseinzadeh, and M. M. Ardakani, *Anal. Bioanal. Electrochem.* 12 (2020) 870.

- [32] D. B. Longley, D. P. Harkin, and P. G. Johnston, *Nat. Rev. Cancer*. 3 (2003) 330.
- [33] R. N. Weinreb, *Ophthalmology* 94 (1987) 564.
- [34] I. A. Alsarra, and M. N. Alarifi, *J. Chromatogr. B* 804 (2004) 435.
- [35] A. R. Buckpitt, and M. R. Boyd, *Anal. Biochem.* 106 (1980) 432.
- [36] A. F. Shojaei, K. Tabatabaeian, S. Shakeri, and F. Karimi, *Sens. Actuators B Chem.* 230 (2016) 607.
- [37] R. Pisano, M. Breda, S. Grassi, and C. A. James, *J. Pharm. Biomed.* 38 (2005) 738.
- [38] S. D. Bukkitgar, and N. P. Shetti, *ChemistrySelect*. 1 (2016) 771.
- [39] M. R. Ganjali, P. Norouzi, F. Faridbod, S. Riahi, J. Ravanshad, J. Tashkhourian, M. Salavati-Niasari, and M. Javaheri, *EEE Sens. J.* 7 (2007) 544.
- [40] H. Karimi-Maleh, C. T. Fakude, N. Mabuba, G. M. Peleyeju, and O. A. Arotiba, *J. Colloid Interface Sci.* 554 (2020) 603.
- [41] M. Baghayeri, B. Mahdavi, Z. Hosseinpor-Mohsen Abadi, and S. Farhadi, *Appl. Organomet. Chem.* 32 (2018) e4057.
- [42] Z. Shamsadin-Azad, M. A. Taher, S. Cheraghi, and H. Karimi-Maleh, *J. Food Meas. Charact.* 13 (2019) 1781.
- [43] M. Baghayeri, M. Rouhi, M. M. Lakouraj, and M. Amiri-Aref, *J. Electroanal. Chem.* 784 (2017) 69.
- [44] A. Khodadadi, E. Faghih-Mirzaei, H. Karimi-Maleh, A. Abbaspourrad, S. Agarwal, and V. K. Gupta, *Sens. Actuators B Chem.* 284 (2019) 568.
- [45] B. Maleki, M. Baghayeri, S. A. J. Abadi, R. Tayebee, and A. Khojastehnezhad, *RSC Adv.* 6 (2016) 96644.
- [46] H. Veisi, F. H. Eshbala, S. Hemmati, and M. Baghayeri, *RSC Adv.* 5 (2015) 10152.
- [47] M. Baghayeri, R. Ansari, M. Nodehi, I. Razavipanah, and H. Veisi, *Microchim. Acta* 185 (2018) 320.
- [48] V. P. Pattar, and S. T. Nandibewoor, *RSC Adv.* 5 (2015) 34292.
- [49] F. Tahernejad-Javazmi, M. Shabani-Nooshabadi, and H. Karimi-Maleh, *Compos. B. Eng.* 172 (2019) 666.
- [50] M. Miraki, H. Karimi-Maleh, M. A. Taher, S. Cheraghi, F. Karimi, S. Agarwal, and V. K. Gupta, *J. Mol. Liq.* 278 (2019) 672.
- [51] M. Abbasghorbani, *J. Mol. Liq.* 266 (2018) 176.
- [52] M. Abbasghorbani, *Int. J. Electrochem. Sci.* 12 (2018) 11656.
- [53] H. Karimi-Maleh, M. Sheikhshoaie, I. Sheikhshoaie, M. Ranjbar, J. Alizadeh, N. W. Maxakato, and A. Abbaspourrad, *New J. Chem.* 43 (2019) 2362.
- [54] A. Faridan, M. Bahmaei, and A. M. Sharif, *Anal. Bioanal. Electrochem.* 12 (2020) 810.
- [55] M. Vahidifar, and Z. Eshaghi, *Anal. Bioanal. Electrochem.* 12 (2020) 712.
- [56] Y. S. Borghei, M. Hosseini, M. Dadmehr, S. Hosseinkhani, M. R. Ganjali, and R. Sheikhnejad, *Anal. Chim. Acta* 904 (2016) 92.

- [57] Y. Orooji, M. H. Irani-nezhad, R. Hassandoost, A. Khataee, S. R. Pouran, and S. W. Joo, *Spectrochim. Acta A* 234 (2020) 118272.
- [58] A. J. Sisi, M. Fathinia, A. Khataee, and Y. Orooji, *J. Mol. Liq.* 308 (2020) 113018.
- [59] Y. Orooji, F. Liang, A. Razmjou, G. Liu, and W. Jin, *Sep. Purif. Technol.* 205 (2018) 273.
- [60] H. Beitollahi, S. Tajik, S. Z. Mohammadi, and M. Baghayeri, *Ionics* 20 (2014) 571.
- [61] H. Rahimi, R. Mozafarinia, R. S. Razavi, E. Paimozd, and A. Hojjati Najafabadi, *Adv. Mat. Res.* 239 (2011) 736.
- [62] Z. W. Ulissi, F. Sen, X. Gong, S. Sen, N. Iverson, A. A. Boghossian, L. C. Godoy, G. N. Wogan, D. Mukhopadhyay, and M. S. Strano, *Nano Lett.* 4 (2014) 4887.
- [63] P. Norouzi, F. Faridbod, B. Larijani, and M. R. Ganjali, *Int. J. Electrochem. Sci.* 5 (2010) 1213.
- [64] S. Ertan, F. Şen, S. Şen, and G. Gökağaç, *J. Nanopart. Res.* 4 (2012) 922.
- [65] H. Karimi-Maleh, F. Karimi, Y. Orooji, G. Mansouri, A. Razmjou, A. Aygun, and F. Sen, *Sci. Rep.* 10 (2020) 11699.
- [66] M. Bijad, H. Karimi-Maleh, M. Farsi, and S. A. Shahidi, *J. Food Meas. Charact.* 12 (2018) 634.
- [67] M. Bijad, H. Karimi-Maleh, and M. A. Khalilzadeh, *Food Anal. Methods* 6 (2013) 1639.
- [68] A. Baghizadeh, H. Karimi-Maleh, Z. Khoshnama, A. Hassankhani, and M. Abbasghorbani, *Food Anal. Methods* 8 (2015) 549.
- [69] T. Jamali, H. Karimi-Maleh, and M. A. Khalilzadeh, *LWT - Food Sci. Technol.* 57 (2014) 679.
- [70] F. Tahernejad-Javazmi, M. Shabani-Nooshabadi, and H. Karimi-Maleh, *Talanta* 176 (2018) 208.