UNIVERSITI TEKNOLOGI MARA

MESOPOROUS SILICA SBA-15 AND SBA-16 AS WORKING ELECTRODE FOR ELECTROCHEMICAL DETERMINATION OF ASCORBIC ACID AND URIC ACID IN WATER

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Thesis submitted in fulfillment
of the requirements for the degree of
Master of Science
(Applied Chemistry)

Faculty of Applied Science

January 2019

ABSTRACT

Mesoporous silica SBA-15 and SBA-16 were synthesized via the surfactant templating technique. Two important components in synthesizing mesoporous silica are the silica source and surfactant. The silica source used was tetraorthosilicate (TEOS). The surfactant used were Pluronic P123 for SBA-15 and Pluronic F127 for SBA-16. SBA-15 and SBA-16 have been proposed as modifiers in carbon paste electrodes for ascorbic acid (AA) and uric acid (UA) detection in water. The physical and chemical characterization of SBA-15 and SBA-16 were carried out using Field Emission Scanning Electron Microscope (FESEM), X-Ray Diffractometer (XRD), Thermogravimetric analysis (TGA), Fourier Transform Infrared (FTIR), and Nitrogen Adsorption. Cyclic voltammetric (CV) method was used for AA and UA electrochemical determination. FESEM analysis showed that the SBA-15 synthesized possessing the rod-like shaped particles. While the SBA-16, exhibits spherical structure. XRD analysis was performed using lower angle which were in between 0.5° to 2.5°. XRD patterns of SBA-15 and SBA-16 showed that the corresponding peaks were indexed at (100),(110),(200) and (110),(200),(211). The surface area isotherms obtained in nitrogen adsorption analysis were both type IV for SBA-15 and SBA-16. Hysteresis loops were observed within the relative pressure range (P/P_o) of 0.45 to 0.70. The average pore diameter for SBA-15 and SBA-16 were 6.01 nm and 6.05 nm. With the application of BJH and BET method, the surface area and pore volume calculated were 531.6 m^2/g and 0.69 cm^3/g for SBA-15. While for SBA-16, it was 626.72 m^2/g and 0.52 cm^3/g. The silica materials became porous after calcination process due to the removal of surfactant. Surfactant in general contains alkyl groups (C-H bonds). Hence, the elimination of C-H stretch band was observed in IR spectrums of calcined SBA-15 and SBA-16, in between the wavelength of 2858 cm^{-1} to 2934 cm^{-1} and 3000cm^{-1}. A curve observed from the dTG graph of as-synthesized SBA-15 in between the temperature of 192°C and 329°C with 30% of mass loss. Together with two separate curves shown between 245°C and 445°C in as-synthesized SBA-16 dTG graphs with approximately 20% mass loss, showing that P123 and F127 was successfully removed during calcination and 550°C was a suitable temperature. SBA-15 and SBA-16 were utilized in the fabrication of carbon paste electrodes; MCPE/SBA-15 and MCPE/SBA-16. The detection limit of MCPE/SBA-15 and MCPE/SBA-16 on AA and UA were successfully identified which are $3.0x10^{-3}$ M and $5.0x10^{-4}$ M, and $-1.0x10^{-4}$ M; $1.0x10^{-5}$ M. The results achieved showed SBA-16 able to promote better enhancement towards AA detection.

ACKNOWLEDGEMENTS

Thank you Allah s.w.t. for giving me the opportunity to even have the chance in pursuing postgraduate studies. That I myself could not believe of having, and others would probably do not even have the chance. The idea of obtaining the scroll after all the struggles and perseverance, I thank Allah swt. with my utmost sincerity for giving me the hope and strength when I am at my lowest and my highest. And I would like to express my heartfelt gratitude to my supervisor, Dr Mohammad Noor Jalil, for the endless guidance and patience with me throughout my journey in completing Master's Degree. Thank you for your kind words, never ending support and a lot of advices not only for me but to take it to the world. I will definitely treasure the priceless knowledge that you have given me.

Not to forget, my best friend, Nurul Barakah Ab Rahman. We have been through a lot throughout the same journey. Countless times of ups and downs, demotivation, problems in finishing this off, but I am truly grateful that we are able to get back up, together, to keep each other from straying too far from the main road. Thank you so much. You have no idea of how much I appreciate your existence, as one of my greatest friends. To the laboratory officers, you are not forgotten when you have helped a lot, especially Mr. Khairul and Mr. Zul from the general chemistry laboratory. Thank you for the willingness of helping me when I need more information about how the instrument works and spare me few of the common chemicals when I am in need. Thank you very much.

Last but not least, I would not be able to end this journey without my parents. They have done so much for me, and how can one repay their parents, the most precious gifts you could ever hope for. Thank you so much, and this one is for you mom and dad. Mr. Mohamad Hassan Abd Wahab and [redacted]

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CHAPTER ONE
INTRODUCTION

1.1 Research Background

Electrochemical determination technique is known as the oldest measurement method comprises a very broad range of possibilities in analytical, enhanced by recent technologies in signal processing [1]. Owing to its effectiveness in the chemical and biological system, electrochemical determination strategies have multiple the intense interest in the community as they have the capacity to be served as the ideal sensing technology. The basic electrochemistry concept implies the transfer charge between an electrode and the liquid or solid phase. Prior to the advanced technology nowadays, electrodes or sensors are mostly being made with higher sensitivity, miniaturized, and low cost [2].

In general, electrodes is basically created as a smart network that acts as one of the important component in the multifunctional integrated electrical devices [3] in which the development of electrodes reveal interesting perspectives in various techniques. Electrodes have been commonly applied in analytical and separation techniques according to published papers such as liquid chromatography, mass spectrometry, electrophoresis, micro-dialysis and electrochemical detection as well [4]. Electrochemical detection techniques are widely known as it is the most rapid, straightforward and cost-effective for detection of compounds [5]. To date, electrodes are functionalized, chemically modified, and enhanced with various types of materials.

There are quite a lot of numbers of researchers worldwide testing the electrode materials including carbon, metal and composite materials. Apart from selecting the perfect base for fabricating electrodes, the modifier is also a contributing factor to the development of electrodes. Modifier is commonly known to compliment the electrode material used in enhancing the effectiveness. Recent studies are found where researchers are fabricating electrodes utilizing in certain type of materials to achieve the surface enhancement effect in detecting analytes. The common analytes applied are often involving favoured molecules [2], heavy metals [6], organic compounds [7] and even humidity [8].