NaSTech
JOURNAL OF NATURAL SCIENCES AND TECHNOLOGIES
2022, 1(1), pp. 135-142, DOI: 10.5281/zenodo.7380122

PREPERATION OF MCM-48 LOADED WITH LAVENDER OIL AND VITAMIN E UNDER SUPERCRITICAL CARBONDIOXIDE CONDITIONS AND ITS USE IN PEEL FORMULATION

Derya KILIÇ[1]*, Emine KUTLU[1], Muhammad Asım ALİ[1], Fatih Mehmet EMEN[1], Ruken Esra DEMİRDÖĞEN[2]

[1]Department of Chemistry, Faculty of Arts and Science, Burdur Mehmet Akif Ersoy University, TR 15030, Burdur, Turkey
[2]Department of Chemistry, Faculty of Science, Çankırı Karatekin University, TR 18100, Çankırı, Turkey

ABSTRACT

This study aimed to prepare a new peeling formulation in which mesoporous MCM-48 loaded with lavender oil and vitamin E in a supercritical carbon dioxide (sC-CO_2) environment is used as a peeling agent. This study targeted at preparing a new peeling formulation which consists of a mesoporous MCM-48 onto which an essential oil such as lavender oil was loaded via a green method that utilized supercritical carbon dioxide (sC-CO_2). This study aimed to prepare a new peel formulation consisting of a mesoporous MCM-48 loaded with lavender oil using supercritical carbon dioxide (sC-CO_2). Structural characterization of MCM-48 and MCM-48-Lavender oil (MCM-48-L) were made via XRD and FT-IR techniques. The XRD powder diffraction pattern shows the amorphous SiO_2 structure at $2\theta=20^o$. Their particle sizes and morphologies were investigated by FE-SEM. The average particle diameter was determined as 590 nm. Pore volumes were determined by N_2 adsorption/desorption analysis. The BET surface area for the prepared MCM-48 was calculated as 2641 m^2/g and the pore diameter of MCM-48 was found to be 3.60 nm. All the results obtained show that MCM-48 loaded with lavender oil and vitamin e can be used as an exfoliating agent.

Keywords: MCM-48, Lavender oil, Chemoexfoliation, Chemical Peeling, Cosmetics.

1. INTRODUCTION

The technique that involves peeling the skin tissue to reach the desired skin depth using chemicals is called chemoexfoliation-peeling. The peeling process helps the skin look cleaner and healthier. This technique is used to evenly remove damaged dermal skin and rejuvenate the skin, as well as minimizing complications such as scarring and unwanted pigment changes [1]. Chemical peels are among the most commonly used cosmetic procedures. Controlled destruction of epidermis and dermis occurs by applying chemical agents on the skin. The skin is induced and exfoliated. Later, dermal and epidermal regeneration observed in the adjacent epithelium and skin appendages causes the surface texture and appearance of the skin to improve [2]. Lavender essential oils have been used in cosmetic and medicinal preparations since Greek and Roman times. Studies have mainly focused on linalool and linalyl acetate, which are found in lavender essential oils, but less common essential oil components (eg, camphor, 1,8-cineol, carvacrol, etc.) are also considered. Among the lavender species, the most cultivated and researched species are *L. angustifolia Mill*., *L. latifolia Medik*. and *L. x intermedia Emeric ex Loisel*, *L. stoechas L*. and *L.luisieri Rozeira* [3, 4]. Many cultivars have been produced by standard breeding experiments [3]. By using metabolic engineering and other techniques, regionally resistant and higher oil yield species are tried to be produced [3-7]. Vitamin E, which has important antioxidant functions especially in cell membranes and lipoproteins, is an important fat-soluble antioxidant. It is frequently used in the cosmetic industry because it nourishes the skin and adds shine. Due to its high antioxidant and protective activity, vitamin E is applied in skin cream formulations, various photo-aging cosmetics and to improve the skin barrier [8]. In 1992, the researchers of Mobil Company developed a series of porous materials and named it as the Mobile Composition of Matter (MCM). MCM-48 (Mobile Compound of Matter No. 48), one of the two most popular mesoporous molecular sieve structures, still attracts the attention of researchers. Composed of amorphous silica walls, the MCM-48 has a long-range regular frame with uniform pores. In addition, MCM-48 has a large surface area, which can be more than 1000 m^2g^{-1}. Moreover, the pore diameter of these materials can be adjusted from 1.5 to 20 nm in a controlled manner by adjusting the synthesis conditions. There are many studies in which mesoporous materials are used as catalysts for various chemical reactions, as a support for a drug delivery system, and as an adsorbent in wastewater treatment [9]. No peeling formulation was found using MCM-48 loaded with lavender oil.

In this study, it was aimed to prepare MCM-48 loaded with lavender oil as the material of the new peel and use it in cream peeling. First, MCM-48 was synthesized and its structural characterization was carried out by XRD and FT-IR techniques. Lavender oil (Lavandula angustifolia) loading into the MCM-48 was carried out in a supercritical carbon dioxide environment. Their particle sizes and morphologies were investigated by FE-SEM. Pore volumes were determined by N_2 adsorption/desorption analysis.

2. MATERIAL AND METHOD

FT-IR spectra were recorded in the 4000-400 cm^{-1} range by Perkin Elmer Fronter FT-IR spectrometer using KBr pellets. X-ray powder diffraction (XRD) using a Bruker AXSD 8 Advance Powder Diffractometer with CuKα = 1.54 Å radiation was used for structural phase identification of MCM-48. Rigaku SMARTLAB instrument was used in low angle XRD measurements. Surface morphologies of MCM-48 was examined via a Jeol Sem-7100-EDX Computer Controlled Digital Scanning Electron Microscope. Nitrogen absorption desorption studies were performed using the Quantachrome Quadrasorb SI system.

2.1. Synthesis of MCM-48

MCM-48 synthesis was carried out according to the method given in the literature [10]. 30 g of CTAB (surfactant) was dissolved in 147 mL of water. Then, about 2.72 g of sodium hydroxide was added to the mixture and stirred for 15 minutes. 30 mL of TEOS was added. The mixture was then stirred for another 3 hours after the temperature was adjusted to about 40 °C. This mixture poured onto a Teflon reactor and heated at 110 °C for 24 hours. Finally, the prepared composites were separated by filtration, washed, and calcined at approximately 550 °C for 6 hours to confirm successful removal of the used CTAB template [10].

2.2. Preparation of the Lavender Oil and Vitamin E Loaded MCM-48

0.5 g of MCM-48 was dissolved in 5 mL of ethanol. 0.5 g lavender oil and 0.5 mL vitamin E were taken into the $scCO_2$ reaction unit heated to 40 ºC. Then it was kept under 200 bar CO_2 pressure for 2 hours. The pressure was reduced by opening the valve of the reaction unit. The lavender oil and vitamin E loaded MCM-48 (MCM-48/L) nanostructure was removed from the unit in a dry state [11].

2.3. Preperation of cream peeling

Cream peeling was prepared using Ceteareth 20, Lanette O, Olea Europaea Fruit Oil, Caprıylyl methicone, PEG-12 Dimethycone, PPG-20 crosspolymer, Unilactamin L7, Panthenol, sodium polyacrylate, EDTA, Phenoxyethanol and lavender oil-vitamin E loaded MCM-48.

2.4. Microbiological Activity Studies

"Total mesophilic aerobic bacteria count" and "Total Yeast-Mold count" analyzes were performed on the peeling sample. The peeling sample was diluted 1/10 in suspension containing 0.09% NaCl. For total mesophilic aerobic bacteria count, PCA (Plate Count Agar) and for Yeast-Mold count PDA (Patato Dextrose Agar) agar medium, the peeling sample, which was diluted with 3 repetitions, was inoculated with 1 ml drop and inoculated in the form of smear sowing. Incubation was carried out at 35 ºC for 48 hours for total mesophilic aerobic bacteria count, and for 72 hours at 25 ºC for Yeast-Mold Counting. Colony count was performed after incubation.

3. RESULTS AND DISCUSSION

3.1. Structural characterization

The structural characterization studies of the MCM-48 were carried out by XRD and low angle XRD dust pattern measurements (given in the XRD graph), and the powder diffraction patterns are given in Figure 1. The XRD powder diffraction pattern shows the amorphous SiO_2 structure at 2θ=20º. The (2 1 1) reflection observed at 3º in the low angle powder diffraction pattern shows the regular mesoporous MCM-48 structure [10].

2022, 1(1), pp. 135-142, DOI: 10.5281/zenodo.7380122

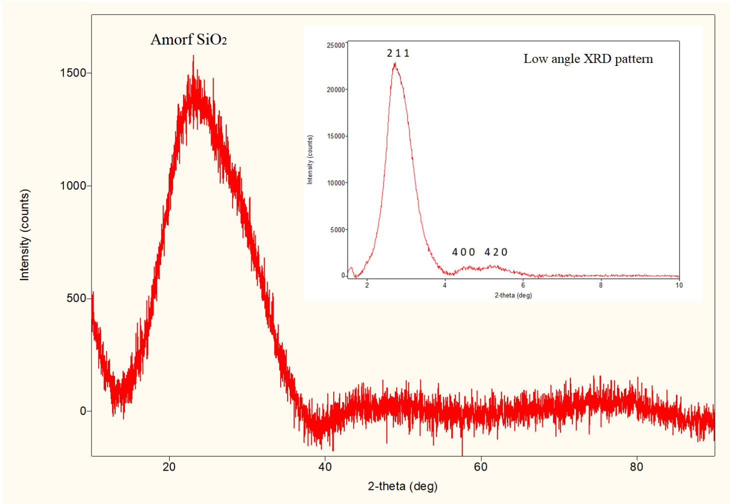

Figure 1. The XRD powder patterns of mesoporous MCM-48

3.2. FT-IR studies

The FT-IR spectra of MCM-48 and MCM-48/L is given in Figure 2. Stretching and bending vibrations of Si-O in MCM-48 were observed at 1050 cm^{-1} and 808 cm^{-1}, respectively. Stretching and bending bands of Si-O in MCM-48/L were observed at 1040 cm^{-1} and 807 cm^{-1}, respectively. The expected C–H stretching bands of 1480, 2854, and 2925 cm^{-1} of CTAB cannot observed in MCM-48 due to removal of surfactants by calcination. The symmetric and asymmetric aliphatic C-H stretching bands are observed at 2861 cm^{-1} and 2932 cm^{-1} in MCM-48/L. This bands can be attributed to the alkyl groups of lavender oil and vitamin E. FT-IR results show successful loading of lavender oil and vitamin E into MCM-48.

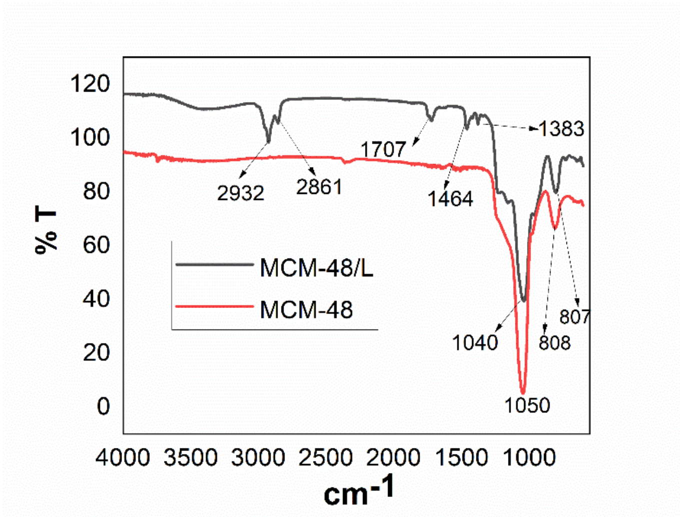

Figure 2. FT-IR Spectrum of the MCM-48

3.3. SEM-EDX Studies

SEM micrographs of MCM-48 are given in Figure 3 and EDX spectrum is given in Figure 4. For SEM measurements, MCM-48 powders, which were turned into pallets, were coated with Pt (containing traces of Pd) and images were taken. It is observed that MCM-48 is obtained in a spherical structure and the particle sizes are homogeneously distributed. The average particle diameter was determined as 590 nm. Elemental analysis of MCM-48 was done via EDX. Si and O atoms were detected as 32% in the EDX spectrum.

NaSTech
JOURNAL OF NATURAL SCIENCES AND TECHNOLOGIES
2022, 1(1), pp. 135-142, DOI: 10.5281/zenodo.7380122

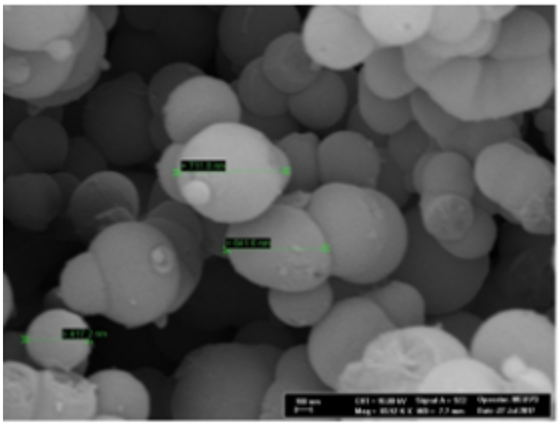

Figure 3. SEM images of the MCM-48

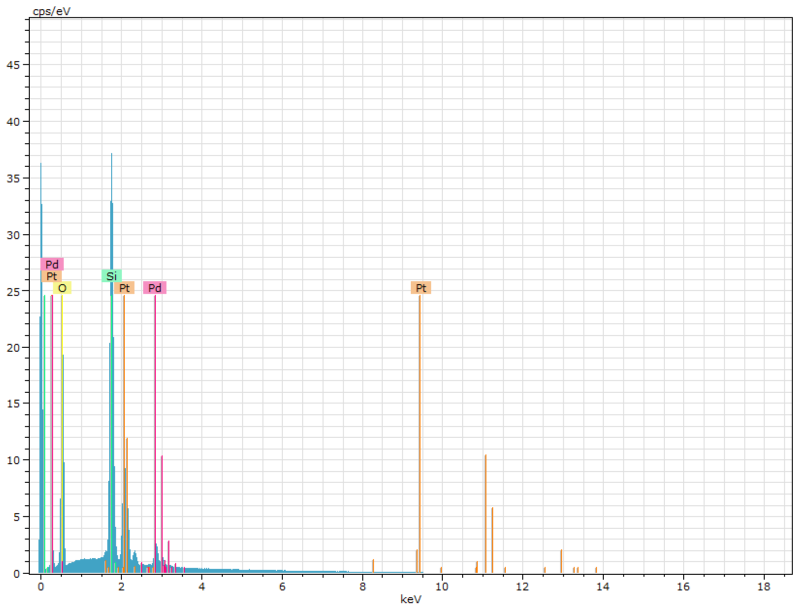

Figure 4. EDX spectrum of MCM-48

3.4. Nitrogen Adsorption/Desorption Studies (BET, BJH)

The pore sizes, pore volumes and surface areas of mesoporous materials are determined using the nitrogen adsorption/desorption technique. According to the IUPAC classification. IV. Type isotherm is associated with mesoporous materials. MCM-48 nitrogen adsorption (blue) desorption (red) isotherms are given in Figure 5. Isotherms IUPAC's IV. It is similar to the type of isotherm. The BET surface area for the prepared MCM-48 was calculated as 2641 m^2/g, which is considerably higher than the 1600 m^2/g value given in the literature. In addition, the pore diameter of MCM-48 was found to be 3.60 nm, in accordance with the literature. These data show that mesoporous MCM-48 with its extremely high area can be prepared. The symmetry of the adsorption/desorption graphs is intact in MCM-48. BET surface areas decreased as expected. An increase was observed in pore volumes and diameters.

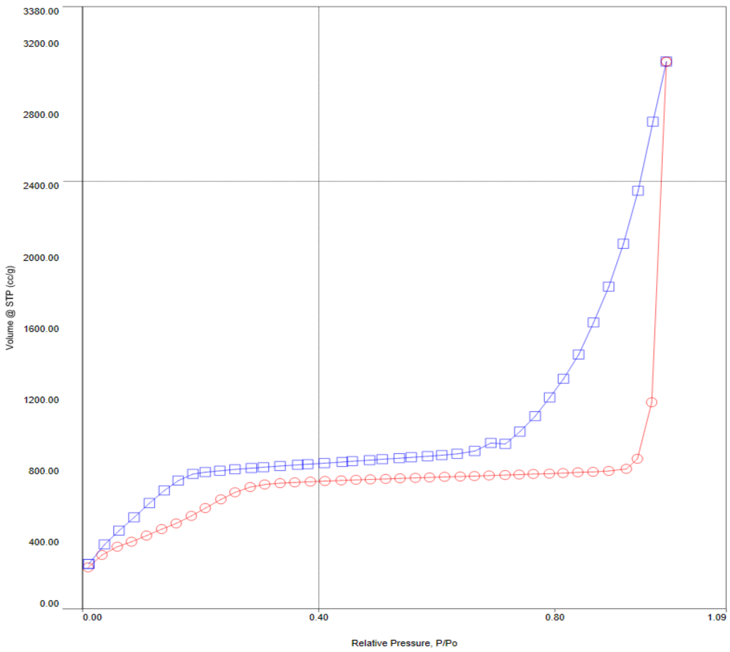

Figure 5. Nitrogen adsorption and desorption isotherms of MCM-48.

3.5. Microbiological Activity Studies

Count analysis of *Staphylococcus aureus, Escherichia coli* and Yeast-Mold microorganisms were performed in the peeling sample. The Biomeriux TEMPO® instrument was studied at 1/40 dilution by applying instrument procedures. Microbiological analysis results are given in Table 1. Microbial activity was not observed in the microbiological analysis which was performed with the "total mesophilic aerobic bacteria count" and "total yeast mold count".

Table 1. Microbiological Analysis Results

Analysis	Kit used	Result
Staphylococcus aureus	TEMPO® STA	< 10 COB/g
Escherichia coli	TEMPO® EC	< 10 COB/g
Yeast-Mold	TEMPO® YM	< 10 COB/g

3.6. Applied of the peeling to the skin

The peeling sample was applied to the hands of two different people, and even in the first application, a visible improvement and brightness was achieved on the skin (Figure 6).

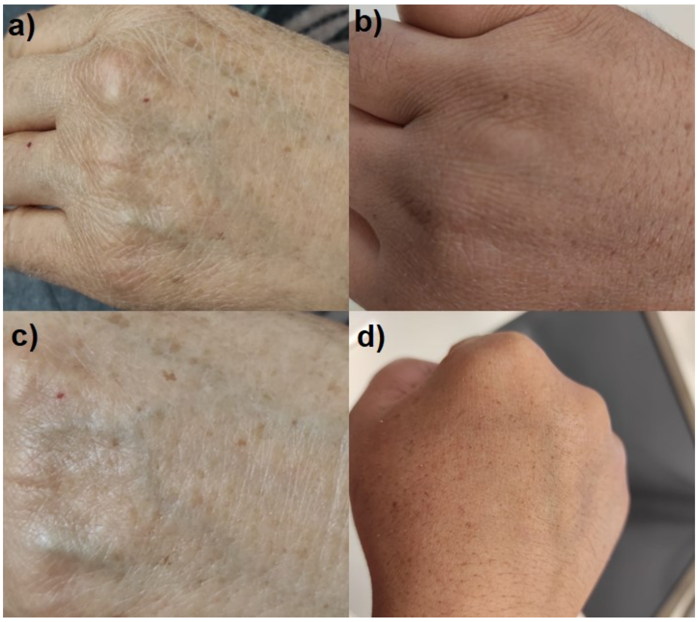

Figure 6. Applied of the peeling to the skin.

4. CONCLUSION

In this study prepared a new peeling formulation which consists of a mesoporous MCM-48 loaded with lavender oil and vitamin via supercritical carbon dioxide (sC-CO_2). Structural characterization of MCM-48 was done by XRD and FT-IR techniques and structural characterization of MCM-48/L nanocomposite was done by FT-IR spectroscopy. The bands observed at 2861 cm^{-1} and 2932 cm^{-1} are aliphatic C-H bands, indicating successful loading of lavender oil and vitamin E into MCM-48. The XRD powder diffraction pattern shows the amorphous SiO_2 structure at $2\theta=20^o$. The (2 1 1) reflection observed at 3^o in the low angle powder diffraction pattern shows the regular mesoporous MCM-48 structure. The pore diameter of MCM-48 was found to be 3.60 nm, in accordance with the literature. PCA (Plate Count Agar) for total mesophilic aerobic bacteria count and PDA (Patato Dextrose Agar) methods were used for Yeast-Mold count in the peeling sample. Microbial activity was not observed in the microbiological analysis which was performed with the “total mesophilic aerobic bacteria count” and “total yeast mold count”. Microbiological analysis results show that there is no reproduction and that the product can be used safely. All the results obtained show that MCM-48 loaded with lavender oil and vitamin e can be used as an exfoliating agent. Thus, lavender oil and vitamin e will be released slowly, and while the shelf life of the product will increase, it will not lose its effectiveness.

REFERENCES

[1] Soleymani T. Lanoue J. Rahman Z. A practical approach to chemical peels: A review of fundamentals and stepby-step algorithmic protocol for treatment. Journal of Clinical and Aesthetic Dermatology. 2018;11(8). pp. 21-28.

[2] Castillo D. E. Keri J. E. Chemical peels in the treatment of acne: patient selection and perspectives. Clinical, cosmetic and investigational dermatology. 2018. 11. pp. 365.

[3] Lis-Balchin, M. (Ed.). *Lavender: the genus Lavandula*. CRC press. 2002.

[4] Upson T. Andrews S. The genus Lavandula. (1st ed.). Timber Press. USA. 2004.

[5] Mendoza-Poudereux I. Muñoz-Bertomeu J. Navarro A. Arrillaga I. Segura J. Enhanced levels of S-linalool by metabolic engineering of theterpenoid pathway in spike lavender leaves. Metabolic Engineering. 2014;(23). pp. 136–144.

[6] Muñoz-Bertomeu J. Arrillaga I. Ros R. Segura J. Up-regulation of 1-deoxy-D-xylulose-5-phosphate synthase enhances production of essential oils in transgenic spike lavender. Plant Physiology. 2006. 142. pp. 890–900.

[7] Muñoz-Bertomeu J. Sales E. Ros R. Arrillaga I. Segura J. Up-regulation of an N-terminal truncated 3-hydroxy-3-methylglutaryl CoA reductase enhances production of essential oils and sterols in transgenic Lavandula latifolia. Plant Biotechnology Journal. 2007. 5. pp. 746–775.

[8] Eiras F. Amaral M.H. Silva R. Martins E. Lobo J.S. Silva A. C. Characterization and biocompatibility evaluation of cutaneous formulations containing lipid nanoparticles. International Journal of Pharmaceutics. 2017.519(1-2). pp. 373-380.

[9] Beck J. S. Vartuli J. C. Roth W. J. Leonowicz M. E. Kresge C. T. Schmitt, K. D. Schlenker, J. A new family of mesoporous molecular sieves prepared with liquid crystal templates. Journal of the American Chemical Society. 1992. 114(27). pp. 10834-10843.

[10] Yu. Q. Zhuang. R. Yi. H. Gao. W. Zhang. Y. Tang. X. Application of MCM-48 with large specific surface area for VOCs elimination: synthesis and hydrophobic functionalization for highly efficient adsorption. Environmental Science and Pollution Research. 2022. 29(22). pp. 33595-33608.

[11] Demirdogen. R. E. Emen. F. M. Karaçolak. A. I. Kılıç. D. Kutlu. E. Meral. O. Preparation of novel CaMoO4: Eu3+-MCM-41 nanocomposites and their applications and monitoring as drug release systems. Journal of Drug Delivery Science and Technology. 2021. 66. pp. 102792.

[12] Everett D. H. Manual of symbols and terminology for physicochemical quantities and units, appendix II: Definitions, terminology and symbols in colloid and surface chemistry. Pure and Applied Chemistry. 1972. 31(4). pp. 577-638.