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#### SYNTHESIS AND CHARACTERIZATION OF POLYVINYL ALCOHOL/CALCIUM CARBONATE COMPOSITE FILM

# ABSTRACT

Calcium carbonate is widely used as a filler material both in plastics and in the pharmaceutical industry in recent years. In this study, the synthesis and characterization of calcium carbonate reinforced polyvinyl alcohol composite film is provided. The synthesized composite film was characterized by FTIR and UV-VIS spectroscopy, the properties of the film were determined by measuring pH and film thickness. Finally, the morphology of the composite film was analyzed by optical microscope. The results show that the polyvinyl alcohol/calcium carbonate composite film was successfully synthesized. According to the results of the optical microscope the calcium carbonate was homogenously dispersed into the film matrix.

Keywords: Composite Films, Calcium Carbonate, Polyvinyl Alcohol, Alcohol, Calcium

#### 1. INTRODUCTION

The preparation of composites reinforced with fillers attracts great interest due to their wide application areas. In addition, obtained of strong composites with the use of safe, eco-friendly, easily accessible and easily manufactured components with active functional groups is an important issue for eco-friendly production [1]. The use of polyvinyl alcohol (PVA) as a polymer matrix in the production of composite materials is a popular choice due to its biological compatibility [2], excellent chemical and physical properties [3], thermal and chemical stability [4]. At the same time, low-cost, easy-to-prepare, non-carcinogenic, film forming ability and high film flexibility make PVA an excellent host material for composite production [5]. Nano or micro scale fillers are more preferred to enhance the functional performance of film based composite materials. In recent years, researchers have focused on composites which has been to design better quality products using less material with nano or micro scale fillers [6]. Structure of PVA contains plenty of reactive hydroxyl groups. This makes PVA easy good distribution of fillers and thus, homogeneous composite films are obtained [2]. Considering the various fillers, calcium carbonate  $(\mbox{CaCO}_3)$  is one of the most widely used fillers due to its low cost and eco-friendly [7]. It is commonly found in living organisms such as sea shell, coral and sea urchin backbone, egg shells and shells of rocks. Calcium carbonate which is a natural substance, has thermally stable, non-toxic, pH sensitive, biocompatible and a good distribution in aqueous solutions [1]. Due to these advantages, CaCO<sub>3</sub> has a significant

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Bildi Ceran, Ö., Şimşek, B., and Şara, O.N., Physical Sciences (NWSAPS), 3A0096, 2020; 15(2):49-53.



potential as filler for various applications such as paper, paint, plastics, rubber, pharmaceuticals, food, ceramics and biological[6]. Studies in the literature, have demonstrated the importance of using micro size CaCO<sub>3</sub> in polymer matrices such as acrylonitrile-butadienestyrene [8], polyvinyl alcohol [9] for the development of mechanical, thermal and rheological properties and in biopolymer matrices such as starch [10] for tissue engineering, packaging applications and drug delivery. In the light of this information, in this study CaCO<sub>3</sub> reinforced PVA composite film was synthesized and characterized by Fourier transform infrared spectroscopy (FTIR) and UV-VIS spectroscopy. The surface image was evaluated by optical microscope image and finally the pH and film thickness were measured.

# 2. RESEARCH SIGNIFICANCE

Synthetic composites reinforced with a small amount of filler material have received great attention because they can be applied in a variety of fields including packaging, medical devices and adsorption. CaCO3 is a biocompatible filler material which can be easily reinforced to polymer matrix. Therefore, it is seen as a new approach in producing high performance new materials in many applications, especially in tissue engineering applications.

#### 3. EXPERIMENTAL METHODS-PROCESS

#### 3.1. Materials

Polyvinyl alcohol (Mw  $\sim$  125.000 and 8.0-98.8 mol% hydrolysis) and calcium carbonate (precipitated for analysis) were obtained from Merck Millipore.

# 3.2. Characterization of PVA/CaCO<sub>3</sub> Composite Film

The chemical structure of the composite was investigated by FTIR spectroscopy. In the analysis, Bruker Tensor II device with a wavelength range of 400 to 4000 cm<sup>-1</sup> was used. The optical transmittance of the composite was recorded with the UV-VIS spectrophotometer in the wavelength range of 200 nm to 800 nm. Surface image of the composite film was obtained using the Nikon SMZ800 stereoscopic zoom microscope. pH value and film thickness were measured with Thermo Scientific Orion device and digital micrometer, respectively.

#### 3.3. Synthesis of PVA/CaCO<sub>3</sub> Composite Film

24 mg  $CaCO_3$  is added in 20 ml deionized water and allowed to mix in ultrasonic bath for 3 hours. 1.2 g PVA is dissolved in 40 ml deionized water at 80°C for 1 hour in a magnetic stirrer. The stirred  $CaCO_3$  solution is added to the dissolved PVA at the end of 3 hours and after 1 drop of glycerin is added, it is allowed to mix for another 1 hour. Before the obtained composite is poured into the petri dish, the pH value is measured. Then the 20 ml of composite is poured into petri dish and allowed to dry at room temperature for 3 days. The experimental setup is shown in Figure 1. Bildi Ceran, Ö., Şimşek, B., and Şara, O.N., Physical Sciences (NWSAPS), 3A0096, 2020; 15(2):49-53.





Figure 1. a) pH meter, b) Composite film and c) Experimental setup

# 4. FINDINGS AND DISCUSSION

# 4.1. pH and Film Thickness

The pH value of  $PVA/CaCO_3$  composite film was measured 8.47 using pH meter. In addition, film thicknesses were measured at 5 points of the surface and the average thickness was calculated as 75.2µm.

# 4.2. FTIR Characterization

FTIR spectroscopy is useful tool for characterizing thin films [11]. The presence of functional groups in pure CaCO<sub>3</sub> and PVA/CaCO<sub>3</sub> composite film were analyzed by FTIR spectroscopy (Figure 2). CaCO<sub>3</sub> exhibited characteristic bands in region of 1350, 865 and 723 cm<sup>-1</sup>. The peak located at 1350 cm<sup>-1</sup> attributed to C=O stretching vibration, the peaks located at 865 and 723 cm<sup>-1</sup> showed O-C-O stretching vibration [11]. In the FTIR spectrum of the PVA/CaCO<sub>3</sub> composite film, a large and intense band was observed at 3260 cm<sup>-1</sup> depending on the presence of the hydroxyl group in the PVA chain. Moreover, peaks at 2935 and 2900 cm<sup>-1</sup> related to methyl group C-H stretching vibration, peaks located at 1090, 1044, 843 and 600 cm<sup>-1</sup> attributed to C-O stretching vibration, C-O stretching vibration and C-H stretching vibration [11].



 $\label{eq:Wavenumbers (cm^{-1})} \enskip \en$ 

Bildi Ceran, Ö., Şimşek, B., and Şara, O.N., Physical Sciences (NWSAPS), 3A0096, 2020; 15(2):49-53.



# 4.3. UV-VIS Characterization

The UV-VIS spectrum showed an absorption band at the wavelength of about 300 nm (Figure 3). The addition of  $CaCO_3$  decreased the absorbance intensity. It was found that the absorbance of the composite film was also reduced due to the very low absorbance of  $CaCO_3$  [12].



Figure 3. UV-VIS spectrum of PVA/CaCO<sub>3</sub> composite film

# 4.4. Morphological Analysis

Surface image of  $PVA/CaCO_3$  composite film obtained by optical microscope. According to the optical microscope image, it was observed that  $CaCO_3$  was distributed homogeneously in PVA film and formed a compact film matrix with PVA film (Figure 4).



Figure 4. Optical microscope image of  $PVA/CaCO_3$  composite film

#### 5. CONCLUSION AND RECOMMENDATIONS

In this study,  $CaCO_3$  reinforced PVA composite film was successfully synthesized. The characterization of the synthesized composite film was carried out with FTIR and UV-VIS spectroscopy. The results showed that homogeneous and compact composite film was obtained. It was observed that  $CaCO_3$  filling material dispersed in the polymer matrix without creating any clumping.



#### NOTICE

This study was presented as an oral presentation at the 2nd International Eurasian Conference on Biological and Chemical Sciences (EurasianBioChem 2019) on 28-29 June 2019.

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