



# Design and Morphology Characterization of Biopolymer Blend-ZnO Nanocomposites Coated Cu-Ni-Mo-based Steel Foam

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## Abstract

Recent developments have been focused on the fabrication and application of metal-metal oxide nanocomposites coated steel foam for nanomaterials, which can have excellent surface morphology and mechanical properties than conventional materials. In this study, a novel 3 dimensional (3D) biopolymer blend-ZnO nanocomposites coated Cu-Ni-Mo-based steel foam was designed and prepared. The objective of this work was to investigate the deposition of the nanofilm by immersion of the steel foam into a solution containing ZnO nanostructures and to determine the effect of the surface coating of biopolymer blend-ZnO nanocomposites onto the Cu-Ni-Mo-based steel foam. A low-cost and easy-to-use dip-coating method was preferred to obtain uniform and high quality coating layers. With this approach, the nanocoatings were prepared at 25 °C and low contact time ( $\approx 10$  min). X-ray diffraction (XRD), scanning electron microscopy (SEM), and stereo microscope analysis methods were used to demonstrate surface and chemical properties of the tragacanth gum / chitosan blend encapsulated ZnO nanocomposites (TG/CH/ZnO NPs) coated Cu-Ni-Mo based steel foam. According to the SEM and stereo microscope images, the prepared 3D random shape with irregular ZnO NPs on the surface of the Cu-Ni-Mo based steel foam were formed. Furthermore, the mean surface roughness values of uncoated steel foam and TG/CH/ZnO NPs coated steel foam were measured as 4.48  $\mu\text{m}$  and 4.61  $\mu\text{m}$ , respectively. Additionally, the RGB pixel of the SEM micrograph of the coated steel foam was analyzed to investigate the effect of coating materials on the surface. Due to cost-efficient and green fabrication of the nanocoating, it has a significant potential to be a promising nanomaterial in biomedical applications.

**Keywords:** ZnO nanocomposite, Steel foam, Nanocoating.

## Biyopolimer karışım-ZnO Nanokompozit Kaplamalı Cu-Ni-Mo-bazlı Çelik Köpüğün Tasarımı ve Morfoloji Karakterizasyonu

### Öz

Son gelişmeler, nanomalzemeler için geleneksel malzemelere göre mükemmel yüzey morfolojisi ve mekanik özelliklere sahip olabilen metal-metal oksit nanokompozitler kaplı çelik köpüğün üretimi ve uygulamasına odaklanmıştır. Bu çalışmada, Cu-Ni-Mo esaslı yeni bir 3 boyutlu (3B) biyopolimer karışımı-ZnO nanokompozitleri kaplanmış çelik köpük tasarlandı ve hazırlandı. Bu çalışmanın amacı, çelik köpüğün ZnO nanoyapıları içeren bir çözeltiye daldırılmasıyla nanofilmin birikmesini araştırmak ve biyopolimer karışımı-ZnO nanokompozitlerinin yüzey kaplamasının Cu-Ni-Mo bazlı çelik köpük üzerindeki etkisini belirlemektir. Düzgün ve kaliteli kaplama tabakaları elde etmek için düşük maliyetli ve kullanımı kolay bir daldırma kaplama yöntemini tercih edilmiştir. Bu yaklaşımla, nanokaplamalar 25 °C'de ve düşük temas süresinde ( $\approx 10$  dk) hazırlanmıştır. X-ışını kırınımı (XRD), taramalı elektron mikroskobu (SEM) ve stereo mikroskop, kitre sakızı / kitosan karışımı ile kapsüllenmiş ZnO nanokompozitlerinin (TG/CH/ZnO NP'ler) kaplanmış Cu-Ni-Mo bazlı çelik köpüğün yüzey ve kimyasal özelliklerini göstermek için kullanıldı. SEM ve stereo mikroskop görüntülerine göre, Cu-Ni-Mo esaslı çelik köpüğün yüzeyinde düzensiz ZnO NP'leri ile hazırlanan 3 boyutlu rastgele yapı oluşturulmuştur. Ayrıca kaplanmamış çelik köpüğün ve TG/CH/ZnO NPs kaplı çelik köpüğün ortalama yüzey pürüzlülük değerleri sırasıyla 4.48  $\mu\text{m}$  ve 4.61  $\mu\text{m}$  olarak ölçülmüştür. Ayrıca, kaplama malzemelerinin yüzey üzerindeki etkisini araştırmak için kaplanmış çelik köpüğün SEM mikrografının RGB pikselleri analiz edilmiştir. Nanokaplamaların düşük maliyetli ve çevre dostu üretimi nedeniyle, biyomedikal uygulamalarda umut verici bir nanomalzeme olma potansiyeline sahiptir.

**Anahtar Kelimeler:** ZnO nanokompozit, Çelik köpük, Nanokaplama.

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## 1. Introduction

With unique developments in nanocoatings, metal oxide nanocoating plays a key role in creative technologies for multidisciplinary applications (Welegers et al., 2021). Among several metal oxide nanocomposites, ZnO nanocomposites are the most leading materials of current developments with superior properties such as biological, mechanical, optical, and electrochemical properties (Najm, 2021; Yang et al., 2021; Rajkumar and Sarma, 2021; Tariq et al., 2021). In particular, ZnO nanocomposites have been fabricated using different methods such as wet chemical, sonication, and microwave methods to obtain uniform nanomaterials (Wagener et al., 2011; Priya et al., 2020; Javed et al., 2020). Considering biopolymer blends have been used as attractive matrixes in several applications with different advantages such as biodegradability, biocompatibility, antimicrobial, antifungal, low-cost, and good transparency.

Metal foams are a new type of material that was first started to be produced in the 1950s and attracted attention among lightweight materials with the developments in recent years (Banhart, 2001). Among metallic foams, especially steel foams stand out with their low cost, high mechanical and thermal damping, lightness, and specific hardness properties (Smith et al., 2012). For these reasons, steel foams are becoming the most promising class of materials in the scientific and industrial areas. There are many studies in the literature on the production, characterization, and investigation of steel foams' properties (Jain et al, 2020; Sazegaran, 2021; Joshi et al., 2015; Yu et al., 1998; Guo et al., 2016). However, since steel foams are a new type of material, they have properties that need to be investigated and developed. It is necessary to examine the coating properties in order to make the use of this new material more widespread and to improve properties such as corrosion resistance and surface functionality (Smith et al., 2012; Banhart, 2001; Shirzadi et al., 2004). Therefore, Cu-Ni-Mo based steel foam produced by the powder metallurgy method was used as the material to be coated in this research. The powder metallurgy method is a prominent method in the production of metallic foam due to its ease, controllability, and final product quality (Beköz Üllen and Karabulut, 2021). The commercial name of the Cu-Ni-Mo-based steel alloy used in the study is Distaloy AB. This steel is primarily used in automotive parts, as well as in the aerospace and machinery industries (Beköz and Oktay, 2014). In this study, a novel tragacanth gum / chitosan blend encapsulated ZnO nanocomposites (TG/CH/ZnO NPs) coated Cu-Ni-Mo based steel foam was developed. The surface morphology and chemical property of the TG/CH/ZnO NPs coated Cu-Ni-Mo based steel foam were investigated using different techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and stereo microscope techniques.

## 2. Material and Method

### 2.1. Materials

Tragacanth gum (molecular weight:840 kDa) and chitosan (molecular weight:141 kDa) were purchased from Sigma Aldrich Company (Germany). Zincnitrate hexahydrate  $Zn(NO_3)_2 \cdot 6H_2O$ , glacial acetic acid (glacial 100%, pro analysis), and sodium hydroxide (NaOH) were purchased from Merck Company (Germany). The chemical content of Cu-Ni-Mo-based steel powders was 1.75 wt.% Ni, 1.5 wt.% Cu, 0.5 wt.% Mo, and the balanced Fe. Space holder (carbamide), binder (paraffin wax),

lubricants (fine graphite and zinc stearate) were supplied from Merck Company (Germany).

### 2.2. Characterization

The surface characterization and chemical properties of TG/CH/ZnO NPs and nanocomposite coated Cu-Ni-Mo based steel foam were determined using different techniques such as stereo microscope, Scanning Electron Microscopy (SEM, FEI QUANTA 450) with a double-coated, 8 mmW × 20 mL, X-ray diffraction (XRD) with Cu K $\alpha$  radiation at 40 kV and 15 mA. The RGB results were recorded using the smartphone (Casper VIA F20) that had a screen size of 6.55 inches and a screen resolution of 720x1600 pixels.

### 2.3. Preparation of Tragacanth Gum-Chitosan-ZnO Nanocomposites

In previous study, the preparation process of the green ZnO nanocomposites was given (Kolatoglu et al., 2020). The nanostructure was prepared using a sonochemical method at room temperature. 0.025 g of tragacanth gum was dissolved in 12.5 ml of distilled water for 2 h. 0.025 g of chitosan was dissolved in 12.5 ml of glacial acetic acid-water solution (2/48, v/v). Tragacanth gum and chitosan solutions were mixed and 0.1 M of Zn  $(NO_3)_2 \cdot 6H_2O$  solution and 0.2 M of NaOH solution were added in the polymer blend. Then, 0.1 M of NaOH solution was added drop by drop to the solution and sonicated at 25°C for 40 min. The sample was first filtered using a sterile filter with a 0.22  $\mu$ m pore size to obtain ZnO nanocomposites.

### 2.4. Preparation of Cu-Ni-Mo-based Steel Foam

Details on the preparation processes of Cu-Ni-Mo based steel foam are reported in the previous paper (Bekoz and Oktay, 2013). The method briefly consists of 4 main steps: preparation of powders, preparation of green compact, removal of space holders, and sintering. A schematic representation of the preparation of the Cu-Ni-Mo based steel foam is given in Figure 1. Firstly, the powders, binder, lubricant, and space holders were mixed in the turbula type mixer for one hour until the space holders were completely coated. Then, the mixture was compacted with a hydraulic press to obtain a green compact with a diameter of 10 mm and a height of 12 mm. Next, the removal of the space holders in the compact steel structure was done in distilled water at room temperature. Finally, Cu-Ni-Mo-based steel foam was obtained by sintering in a laboratory tube furnace at 1150 °C for 60 minutes.

### 2.5. Preparation of Nanocomposite Coated Cu-Ni-Mo-based Steel Foam

In order to coat the produced steel foams more easily, the cutting process with Electrical Discharge Machining (EDM) was applied. The photograph and process parameters of the Cu-Ni-Mo based steel foam before and after the cutting process are given in Figure 2. After the cutting process was completed, the dust caused by the cutting process on the surface was removed by compressed air. Cu-Ni-Mo-based steel foam pieces sliced in 0.5 cm thickness were dipped in the TG/CH/ZnO NPs solution for 5 min at room temperature and then dried at 50 °C for 3 hours. According to the weight difference results of the uncoated foam before and after coating, 2.95 mg of solution was loaded into the foam.

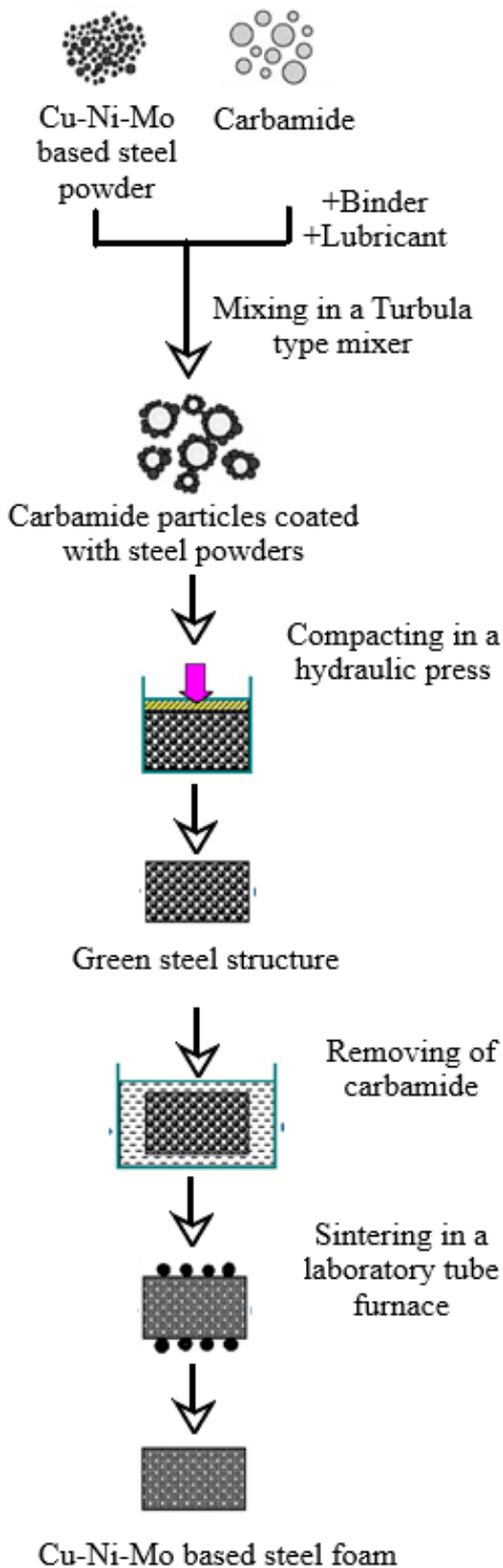


Figure 1. Preparation of Cu-Ni-Mo based steel foam



Figure 2. Photograph of produced and sliced Cu-Ni-Mo based steel foam and cutting process parameters

### 3. Results and Discussion

#### 3.1. Characterization of Tragacanth Gum-Chitosan- ZnO Nanocomposite

In previous study, XRD and SEM techniques were used to demonstrate surface and chemical properties of the TG/CH/ZnO NPs. XRD analysis was used to determine the structural and crystalline phases of TG/CH/ZnO NPs. In Figure 3, SEM micrograph of TG/CH/ZnO NPs revealed the uniform morphology with self-assembled monolayer of prism shaped particles. Furthermore, prepared TG/CH/ZnO NPs have a homogeneous distribution with ZnO particles below 100 nm in size (Kolatoğlu et al., 2020).

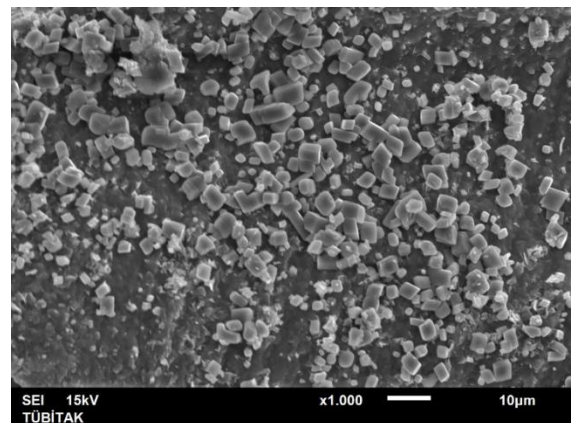


Figure 3. The SEM micrograph of TG/CH/ZnO NPs.

In Figure 4, the prepared TG/CH/ZnO NPs patterns were exhibited three predominant crystalline peaks  $2\theta$  value at  $13^\circ$ ,  $31^\circ$ ,  $35^\circ$  and  $40^\circ$  which indexed to the planes  $\{102\}$ ,  $\{100\}$ ,  $\{002\}$ , and  $\{101\}$ , respectively (JCPDS-897102) (Kolatoğlu et al., 2020). The Scherrer's equation was used to calculate the average crystallite size of the particles (Singh et al, 2016).

$$D = 0.94\lambda/\beta\cos\theta \quad (3.1)$$

Where, D: the crystallite size perpendicular to the reflecting planes,  $\lambda$ : wavelength of X-rays,  $\beta$ : the broadening of diffraction



line at half of its maximum intensity at half maximum, and  $\theta$ : the angle of diffraction. The crystallite size of the ZnO NPs was calculated to be 16.71 nm using the Scherrer's equation.

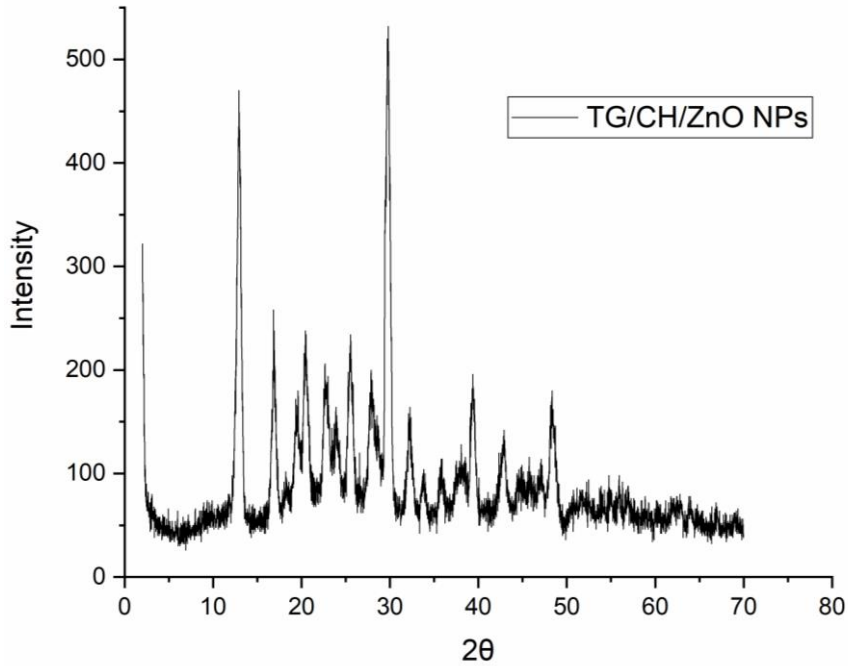


Figure 4. XRD analysis of TG/CH/ZnO NPs.

### 3.2. Characterization of Cu-Ni-Mo-based Steel Foam

Cu-Ni-Mo-based steel foams having about 72% porosity were produced by the powder metallurgy technique. Figure 5 shows the stereo microscope image of pores structures. SEM image of steel foam is shown in Figure 6. The final state morphology of the pores of the sintered Cu-Ni-Mo based steel foams has resembled the initial shape of the carbamide particles. The foam consists of 72% total porosity, with 55% open and 17% closed. The mean equivalent spherical diameter and mean sphericity, examined from the SEM image using image analyzer software, were determined as 612  $\mu\text{m}$  and 0.55, respectively.

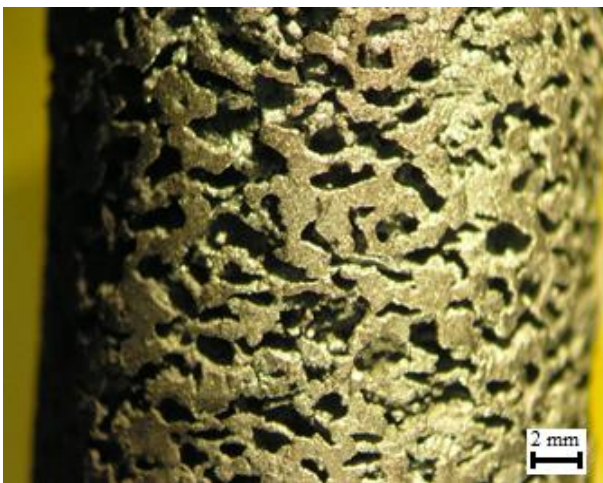


Figure 5. The stereo microscope image of Cu-Ni-Mo based steel foam

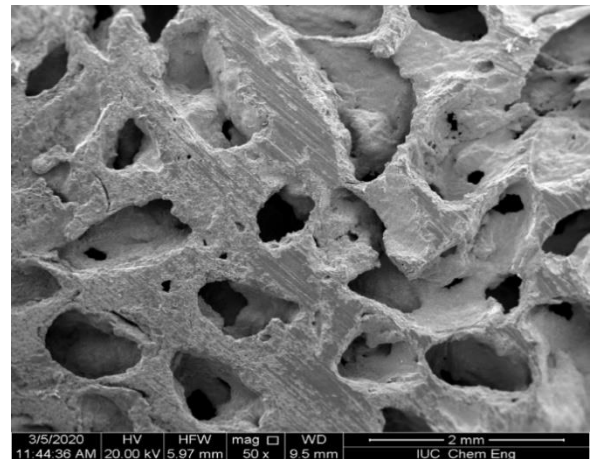


Figure 6. The SEM images of Cu-Ni-Mo based steel foam

### 3.3.Characterization of Nanocomposite Coated Low Alloyed Steel Foam

In order to better understand the post-coating structure, SEM image of the pre-coating structure is given in Figure 7. SEM images of the coated samples at different magnifications are given in Figure 8, Figure 9 and Figure 10. The change in the coated surface of the steel foam compared to the uncoated surface is observed from the images. Looking at the images at larger magnifications, it can be said that the coating structure is in a geometric form with sharp corners. There are a few published reports on coating of stainless steel foams. Karakuş et al. (2020a), studied the surface behavior of ZnO nanoparticles coated 17-4 PH stainless steel foam, and understood from the SEM images that the steel surface is homogeneously coated with the nanostructure,

with the branched structure formed after the coating. In another study of the Karakuş et al. (2021), investigated the surface behavior of coated 17-4 PH stainless steel foam with Kappa-Carrageenan/PVA/ES nanobiocomposite, and similar results have been reported.

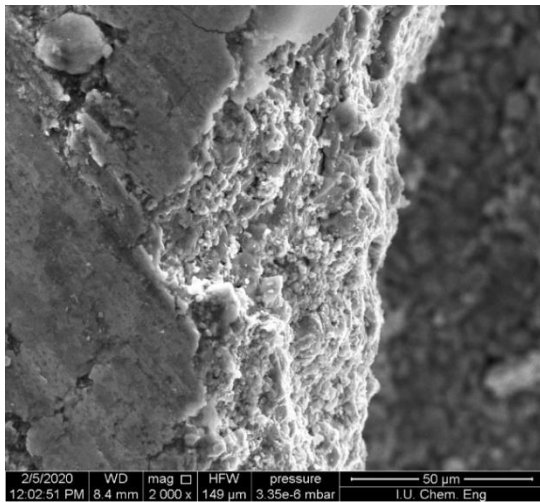


Figure 7. The SEM images of uncoated Cu-Ni-Mo based steel foam

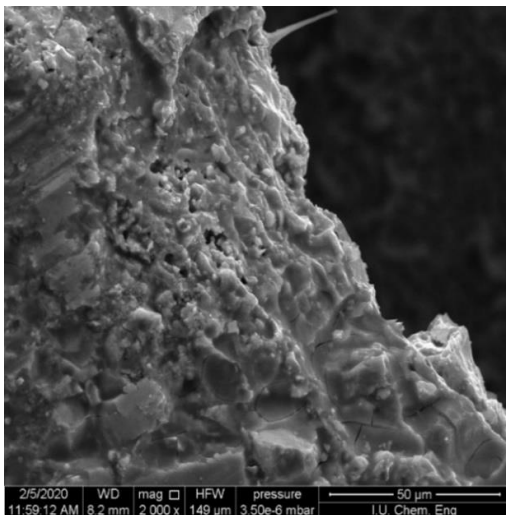


Figure 8. The SEM image of coated surface at x2000 magnification

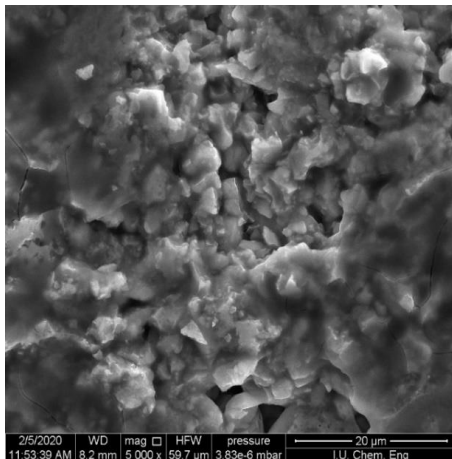


Figure 9. The SEM image of coated surface at x5000 magnification

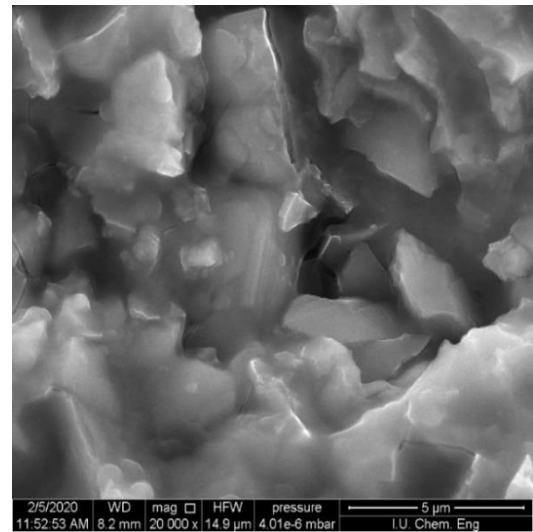


Figure 10. The SEM image of coated surface at x20000 magnification

The mean surface roughness values of uncoated steel foam and TG/CH/ZnO NPs coated steel foam were measured as 4.48  $\mu\text{m}$  and 4.55  $\mu\text{m}$ , respectively. The coating thickness value was measured as approximately 21  $\mu\text{m}$  with an optical microscope. A closer view of the pore walls of the coated steel foams was presented in Figure 11. The presence of very prominent micropores in the cell walls was detected. These micropores affect the properties of the porous structure by providing communication between macropores. No cracks were detected along with the interface during the coating process of the steel foam. The surface properties of coated 17-4 PH stainless-steel foam with CMC/Chitosan- $\alpha$ - $\text{Fe}_2\text{O}_3$  nanoparticles (NPs) was studied by Karakuş et al. (2020b), the authors reported that the average surface roughness and coating thickness values coated stainless steel foam to be between 4.59–5.91  $\mu\text{m}$  and 15 and 30  $\mu\text{m}$ , respectively.

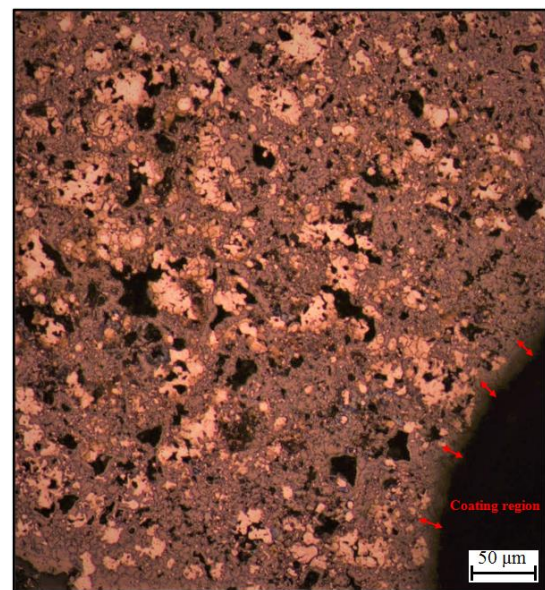


Figure 11. Cross-sectional surface microstructure of the TG/CH/ZnO NPs coating on the Cu-Ni-Mo based steel foam substrate

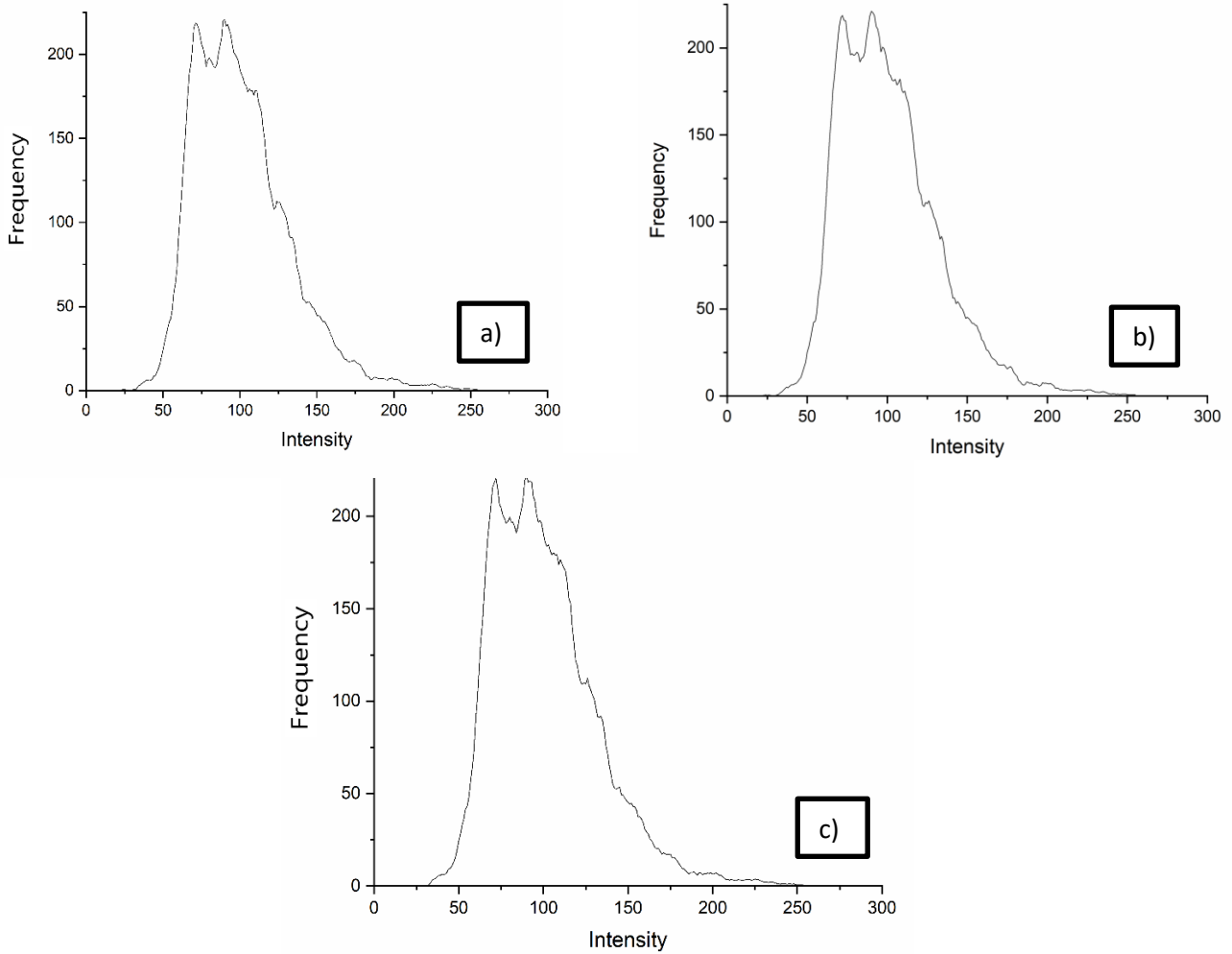


Figure 12. RGB analysis a) red b) green c) blue color of the SEM image of coated surface.

In this study, the RGB pixel of the SEM micrograph of the coated steel foam was analyzed to investigate the effect of coating materials on the surface. The color intensity of RGB image of the coated foam was related to the variation of the recombination probabilities from different areas of the SEM mapped part (Rodriguez et al., 2021). With this approach, it was found that SEM/RGB images exhibited a close result in red (101.01), blue (101.01), and green (100.99) intensities due to the material coating process. Finally, a uniform morphology was observed without a significant sharp increase on the surface of the region, corresponding to the coating and the process successfully happened.

#### 4. Conclusions and Recommendations

Biopolymer blend-ZnO nanocomposite was successfully coated on Cu-Ni-Mo-based steel foam for the first time in this study. The surface property of the prepared biopolymer blend-ZnO nanocomposite was successfully coated on Cu-Ni-Mo-based steel foam was tested using the SEM image based RGB color method. The nanoparticles had a prism shape and the crystal size of the nanostructure was determined as 16.71 nm. It was observed that the shape of the nanoparticles was preserved after the coating, and the

surface was uniformly coated with an average thickness of 21  $\mu\text{m}$ .

The coating characteristic of metal foams is not only affected by the properties of the foam but also depends on the properties of the material to be coated, namely the substrate. The effect of the use of steel powders containing different alloying elements on the coating properties should be investigated. The coating behavior of steel foams has become a very important subject for research with the wide range of applications of these materials. It is hoped that this study will help researchers working on this subject.

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