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Reflection Loss Improvement by Cerium Addition in Chitosan-Hydroxyapatite Film as Stealth Drone Candidate

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Abstract— Radar is a detection and tracking technology commonly applied to monitor environmental conditions. However, their ever-growing capabilities pose a serious challenge to military operations because they increase the risk of being detected by the enemy. Conversely, unmanned aircraft (drones) are increasingly widespread in gathering information. The effectiveness of this technology can be reduced due to exposure to radar waves that allow detection. Therefore, developing coating materials capable of absorbing radar waves is urgently required to increase the effectiveness of military equipment. The composites developed were derived from chitosan obtained from crustacean waste, hydroxyapatite obtained from eggshell waste, and the rare earth metal cerium obtained from Lapindo Mud. Composites containing cerium metal (Ce) demonstrated superior radar signal absorption capabilities than samples without Ce. This result was evidenced by VNA measurements showing increased absorbance in the 100 MHz - 8.5 GHz frequency range. SEM tests indicated that cerium particles enhanced the density and homogeneity of the pore structure, with a size range of 17–24 µm. FTIR characterization revealed that Ce was physically bound to the chitosan-HAp composite. In mechanical testing, the composite with Ce exhibited a maximum tensile stress of 9.512 MPa and a strain of 9.512%, whereas, without the addition of Ce, a stress of 9.529 MPa and a strain of 25.512% was obtained. These findings suggested that incorporating rare-earth metals into chitosan-HAp composites could enhance their ability to absorb radar waves, indicating significant potential for applications in defense technology.

Keywords— *Cerium; Chitosan; Hydroxyapatite; Radar absorbing material*

1. INTRODUCTION

Drones are one of the superior technologies that complement and support military operations [1]. Drones are capable of reconnaissance and attack during war. However, radar systems can still detect drones, making it possible for enemy attacks. It was further stated that China has produced a drone technology with stealth capabilities that can avoid detection by a state protection system. Based on this, innovation is needed to complement the performance of drones in maintaining state defense, namely anti-radar materials that coat drones so that enemy air defense radar systems do not detect them as a strategic step in the development of anti-radar materials, raw materials have abundant availability and economical production costs, per the principles of utility and completeness as stated in Permenhan No. 35 of 2015. Lapindo mud and marine biota waste, such as shrimp shells, are selected as the basic materials for making radar materials. The use of local resources supports the development of conservation technology and contributes to efforts to manage waste more sustainably. Indonesia is known as a maritime country because it has a wider sea area than its land area, thus producing abundant marine resources. However, until now, the use of marine products has been focused on processing meat as food, and the waste has not been optimized optimally.

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One of the high-value compounds extracted from marine biota waste is chitosan. Chitosan is known and developed as a radar wave-absorbing material that is thought to be able to store energy from radar waves. Chitosan has polyelectrolyte properties, can degrade in nature, is non-toxic, is an amorphous solid, and can increase transparency, making it a potential candidate for coating material in stealth applications [2]. Chitosan is generally made from crustaceans, such as shrimp, crabs, insects, algae, and shellfish, which are sources of chitin [3].

Based on data from the Ministry of Maritime Affairs and Fisheries (KKP), Indonesia's total shrimp production in 2020 reached 1,107,264.65 tons, indicating a high shrimp consumption within the community. However, this increase in consumption is directly proportional to the amount of waste produced. If not managed properly, this waste can cause environmental pollution. According to Zam et al. [4], shrimp waste, if not optimally utilized, can cause environmental damage because the resulting shell waste is easily decomposed and difficult to degrade. This disrupts the aesthetics of regional spatial planning and heightens of the risk of pollution. Therefore, using shrimp waste as a raw material for radar wave-absorbing material provides added value to the waste and supports the development of stealth technology [5]. In addition to being polymer-based, chitosan, wave-absorbing materials, and radiation-absorbed materials (RAM) also use metal elements to improve their electromagnetic properties. One of the metals that has a high potential for improving RAM layers' magnetic and electrical properties is cerium (Ce), which is abundant in Indonesia.

The Sidoarjo Hot Mud is an event of hot mud gushing at the Lapindo drilling site owned by PT. Brantas in Balongnongo Hamlet, Renokenongo Village, Porong District, Sidoarjo Regency, East Java, from May 29, 2006 [6]. Based on the research results conducted by Agustawijaya et al. [7], Lapindo mud exhibits a porous smectite structure of shale rock type, an ideal absorbent container. Based on the results of other research on the metal content of Lapindo mud conducted by [8] show an analysis of the emission spectrum of four different types of rare earth metals, namely Yb, Y, Eu, and La. Lapindo mud, which has produced losses and wastes, can contain RAM metal, making Lapindo a source of raw metal extraction in this study.

The urgency of maintaining confidentiality in military movement strategies is extremely high. However, the development of radar wave-absorbing materials incorporated with Cerium is still relatively limited. This study aims to examine the synthesis method for antiradar film layers based on chitosan-hydroxyapatite (HAp) with the addition of cerium metal (Ce) and to evaluate the resulting material's properties and characteristics. In addition, this study aims to analyze the effect of the combination of chitosan-HAp with rare earth metals on increasing the effectiveness of radar wave-absorbing coating material in military equipment. In addition to functioning as a solution for reducing detection by enemy radar systems, this study also utilizes abundant materials that are often considered waste and have no economic value. This study identifies the optimal formulation and explores further developments in the manufacture of radar wave-absorbing materials. By utilizing the abundant rare earth metal Ce from Lapindo mud, Crustacean animal waste as a source of chitosan, and eggshell waste as a source of Hap, this study aims to support the independence of the Indonesian defense industry by optimizing the use of available natural resources.

2. EXPERIMENTAL SECTION

2.1. Materials

The materials used in the research were shrimp waste, egg shells, and Lapindo mud. In addition to the waste, several chemicals were used, including acetic acid 2% No. CAS 64-19-7, sulfuric acid 3 M No. CAS 7664-93-9, oxalic acid No. CAS 144-62-7, nitric acid No. CAS 7697-37-2, tributyl phosphate No. CAS 126-73-8, sodium nitrite No. CAS 7632-00-0, hydrochloric acid 1N No. CAS 7647-01-0, sodium hydroxide No. CAS 1310-73-2, distilled water No. CAS 7732-18-5, disodium phosphate No. CAS 7558-79-4, acetic acid 0.01 M No. CAS 64-19-7, polyvinyl alcohol (PVA) No. CAS 9002-89-5, hydroxyapatite No. CAS 1306-06-5, and cerium oxide No. CAS 1306-38-3.

2.2. Instrumentation

The composite was characterized using Vektor Network Analyzer (VNA), SEM JEOL JSM-6510, FTIR Shimadzu Prestige 21, and tensile strength analysis (Tensile Strength Test).

2.3. Synthesis of Chitosan

In the sample preparation stage, the shrimp shells were washed repeatedly and sun-dried for 4 days. Furthermore, the dried shrimp skin was mashed with a grinder and sieved. Chitosan isolation was carried out through 4 stages: sample preparation, deproteination, demineralization, and deacetylation. 400 g of the sifted powder was weighed and mixed with 4 L of a 3.5% NaOH. The solution was stirred and heated to 65 °C. The heating results were washed with distilled water until the pH was neutral. The mixture was filtered and then oven-dried at 60 °C for 4 h until protein-free dry solids were formed.

2.4. Synthesis of Hydroxyapatite (HAp)

The eggshells were cleaned and dried in the oven for 1 h. Once dry, the egg shells were ground into powder.

The shell powder was sieved until a powder measuring about 100 mesh was obtained. Furthermore, the CaCO3 content of the eggshells was calcinated into CaO. The existing porcelain cup was rinsed using concentrated HNO_3; then, the eggshell powder was put in the cup and heated in the furnace at 900 °C for 3 h. The calcination results were then air-dried for a week at room temperature.

2.5. Metal Extraction from Lapindo Mud

The soil samples were cleaned using 3 M H2SO4 with a ratio of 1:50 (w/v) referring to Sinha et al. [9], then centrifuged for 1 h. The filtration produced residue; the resulting filtrate solution was precipitated using oxalic acid within a few minutes. The resulting precipitate was calcined at 100 °C. The results of the calcination were carried out a second washing using HNO_3, and then extracted using the TBP method which refers to Maiti et al. [10]. The extracts in the form of organic compounds were taken, and then stripped using $NaNO_2$ solution, where the aqueous phase obtained was Ce.

2.6. Composite Synthesis

The composite was prepared from chitosan containing hydroxyapatite, and cerium was added to several samples. The chitosan solution was prepared by dissolving 2 g in 100 mL of 1% acetic acid, whereas the PVA solution was prepared by dissolving 5 g of PVA in 100 mL of distilled water at 90 °C. Furthermore, both solutions were allowed to stand until the temperature was ± 25 °C, and then homogenized using a hot magnetic stirrer for 10 min. 0.5 gram of HAp and 1 g of Ce metal were added. Then, homogenized again for 10 min. This test refers to Irianto et al. [11].

3. RESULT AND DISCUSSION

3.1. Product Description

The product created is an anti-radar film composed of chitosan-HAp and the rare earth metal Ce. This film layer can absorb radar waves, as evidenced by testing using the Vector Network Analyzer (VNA) instrument. In addition, this product exhibits an enormous tensile strength, demonstrated by the Shimadzu AGS 10kN Universal Testing Machine (UTM) test instrument. Due to its relatively strong tensile strength and great elasticity, this film can be applied to drones as an innovative Stealth-Drone solution that can absorb radar waves, so that enemies cannot detect them. The advantage of this product is the abundance of materials originating from waste, an environmental problem in Indonesia, which can be turned into supporting material for defense technology. The fast and easy manufacturing process is an advantage of this product. This product upholds the principles of efficiency and economy by Minister of Defense Number 35 of 2015 concerning the Implementation of Requirement Planning for the Main Equipment of the Indonesian National Armed Forces Weapon System within the Ministry of Defense and the Indonesian National Armed Forces. The visualization of the composite can be seen in **Fig. 1**.

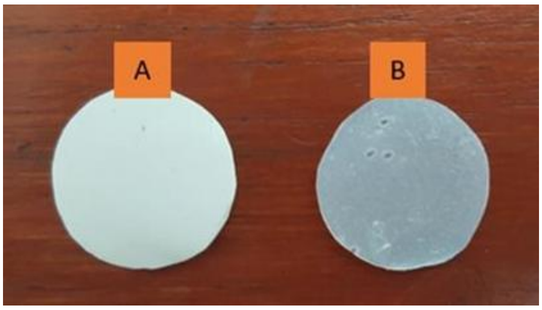

Fig. 1. (A) Chitosan-HAp-Ce composite, and (B) Chitosan-HAp composite.

3.2. Characterization of the Composite Film Layer

The characterization involved two samples: sample A (film layer of chitosan-HAp composite and Ce metal) and sample B (film layer of chitosan-HAp composite) (**Fig. 1**). These two samples were compared to determine the effect of adding Ce metal and to evaluate their potential effectiveness radar-absorbing materials for drones.

Reflection loss is an analytical method used to determine how much absorption of electromagnetic waves (radar) by the material made. This test was carried out in the 100 KHz–8.5 GHz range. Radar wave-absorbing material Based on the VNA test results in **Fig. 2**, the graph shows that the more negative the absorbance value, the greater the power absorbed, so the reflected signal was smaller.

It can be observed that the film layer without Ce metal exhibited a smaller reflection loss value than the film layer with Ce metal. This indicates that Ce metal content can increase the absorption of radar waves in the sample. This occurs because Ce metal can act as a Radar Absorbing Material (RAM), causing waves that enter the RAM material to collide with the particles in the material. Because of this collision, the waves lose energy and are not reflected [12]. The thickness of the

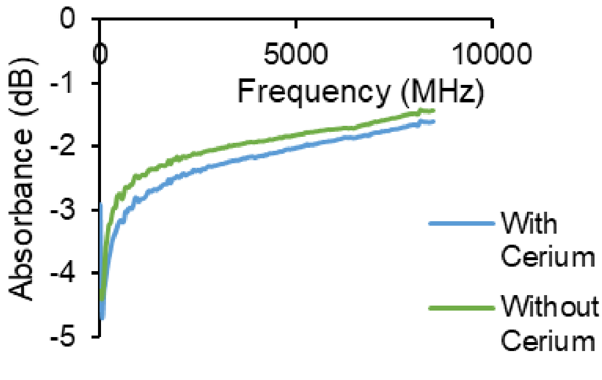

Fig. 2. VNA test result of composite

film layer is very influential on the results of the radar absorption test. The thicker the film coating, the greater the reflection loss value [12]. The materials used in the manufacture of prototypes are obtained from natural synthesis. Therefore, there is a need for further optimization in the manufacture of precursors to achieve optimal radar absorption values.

SEM analysis is used to determine the morphology of the prototype radar-absorbing material. SEM produces better images than light or optical microscopy (MO). This observation was performed on the horizontal section, the top surface, and the cross-section.

The SEM characterization of the sample is presented in **Fig. 3A**, which represented a prototype with a mixture of Ce metal. This result demonstrates that the materials used can be mixed well, thereby forming an interaction in the composite mixture with Ce metal. This homogeneity affects the product's durability, strength, and flexibility to be applied [13]. A more homogeneous mixture generally exhibited greater resistance and strength than a less homogeneous mixture.

Fig. 3B shows that the prototype material with the Ce mixture has a more compact and homogeneous composition than that of with numerous cavities. This homogeneity is indicated by the shape and colour of the crystals, which tend to be uniform. The film layer on a flat surface had a fairly good level of evenness, but lumps could be seen on the film surface. This result indicates incomplete homogenization. The -OH group in Chitosan can affect its mechanical properties and film solubility. Increasing the gelatine concentration increased the tensile strength but reduced the water solubility of the composite film. Films containing 23.5% gelatine showed the highest tensile strength [14].

A tensile strength analysis is carried out to determine the strength of the prototype. The samples whose tensile strength was analyzed were A and B. The tensile strength values of sample A and sample B at the same thickness were 13.66 MPa and 9.529 MPa, respectively. This result proves that the presence of Ce metal in the sample produces stronger resistance compared to the absence of Ce metal. The results of the tensile strength analysis are presented in **Fig. 4A.**

The strength of the tensile test was illustrated from the SEM test of composite sample A containing Ce, which had a denser structure, such that the PVA used as a binder binds strongly at a closer distance between the particles. The arrangement of the small cavities showed many intermolecular bonds, contributing to its high strength. Therefore, it can be concluded that the tensile strength of sample A is quite large compared to that of sample B because it has a more porous structure and greater spacing between its particles.

The elasticity test (**Fig. 4B**) revealed that sample A could elongate up to 9.521%, while sample B could elongate up to 25.58%. These results indicate that sample B elongation ability is greater than sample A. The visible structures observed in the SEM test results

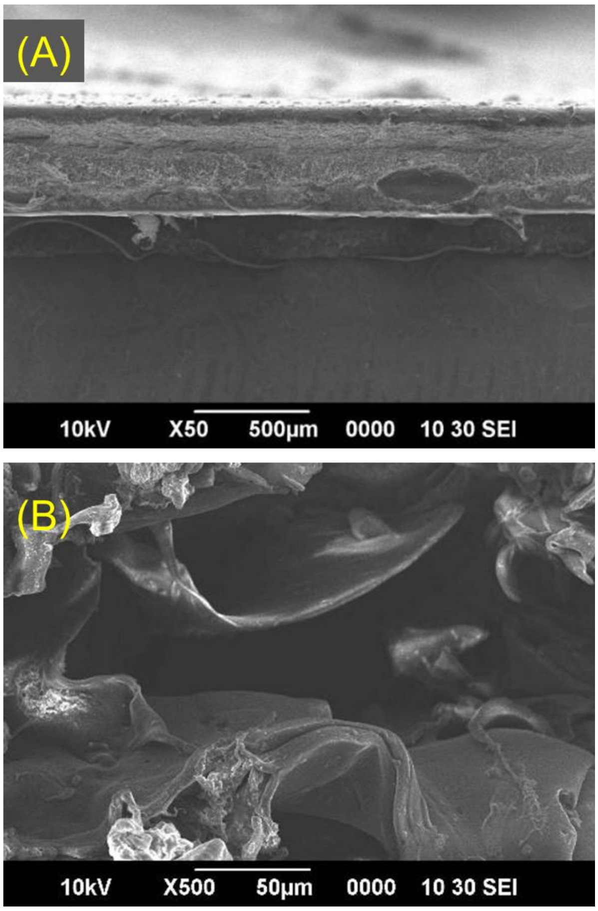

Fig. 3. SEM image of Chitosan-HAp-Ce composite at (A) 50 and (B) 500 magnitude.

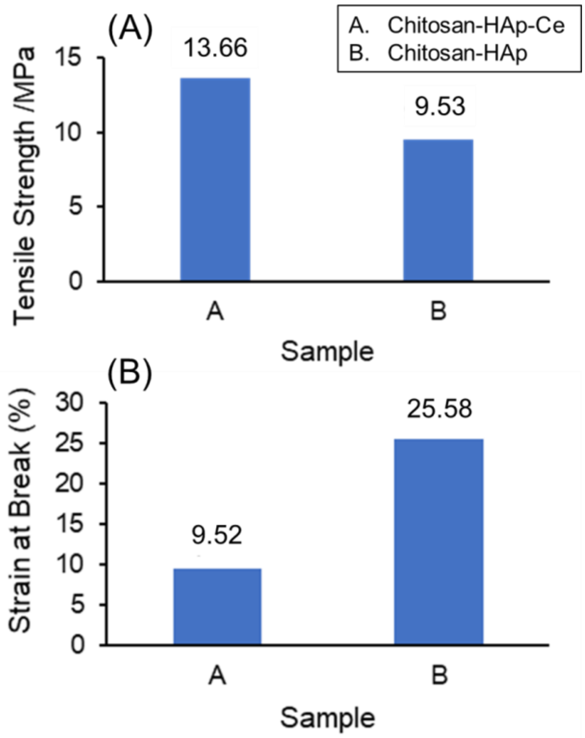

Fig. 4. (A) Tensile strength result and (B) elasticity test result of Chitosan-Hap-Ce and Chitosan-Hap.

illustrated the composite's elastic properties. A large cavity are associated with large tensile capacities, whereas a small cavity correspond to lower tensile capacities [15]. Based on the SEM test, sample B had a larger cavity diameter than that of sample A. The Ce metal filling the voids in sample A decreased the number and size of the pores and increased the density of the particles. According to Dadzie et al. [16], the higher the particle density, the higher the toughness. Therefore, sample A, which had a higher particle density, showed a more firm or rigid composite structure, ultimately leading to lower elasticity compared to sample B.

Based on the FTIR spectrum (**Fig. 5**), HAp was confirmed by the absorption peaks observed at 1023.47 and 1416.95 cm^{-1}. The peaks indicate the presence of carbonate ions (CO_3^{2-}) and phosphate groups (PO_4^{3-}), which are characteristic features of HAp. In addition, chitosan can be identified through the absorption peak at 1586.38 cm^{-1}, representing the bending of $-NH_2$ groups (amine groups in chitosan). Furthermore, the absorption peak at 2840 cm^{-1} indicates the presence of aliphatic -CH groups from both chitosan and PVA.

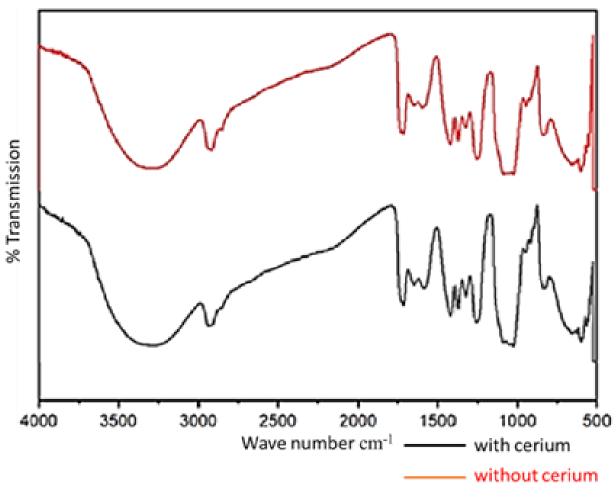

Fig. 5. Fucntional group analysis by FT-IR

The analysis results presented in **Fig. 5** indicate that the functional groups of each sample did not have a significant difference. Alteration in the sample's wave number of functional groups can occur due to interactions among mixed material components [17]. In the samples that contained chitosan-HAp and Ce, no chemical bonds were formed between the constituent compositions, as evidenced by the absence of new peaks in the FTIR analysis [18]. The findings suggest that Ce is physically bound to the chitosan-HAp composite, as no significant differences in the functional group peaks are observed in the FTIR spectrum. The physical interactions may involve mechanisms, such as electrostatic attraction, hydrogen bonding, or van der Waals forces, as well as the formation of strong covalent bonds. Ce's presence may affect the composite's structural properties by improving its homogeneity, mechanical strength, and radar absorption capabilities.

CONCLUSION

This study successfully developed a radar wave-absorbing material based on chitosan-hydroxyapatite (HAp) composite with the addition of cerium (Ce) metal (CeCHap). Incorporating cerium enhanced the material's morphological structure, increasing its density and cohesiveness, and improving mechanical properties. The CeCHap film exhibited a higher tensile strength (13.66 MPa) than the composite without cerium (9.529 MPa). Additionally, vector network analyzer (VNA) measurements confirmed that the CeCHap film effectively absorbed radar waves across the 100 KHz – 8.5 GHz frequency range, demonstrating its potential for stealth technology applications.

The use of locally sourced materials, including chitosan from crustacean waste, hydroxyapatite from eggshells, and cerium extracted from Lapindo mud, highlighted the strategic advantage of this research in supporting sustainable resource management while enhancing national defense capabilities. Given its effectiveness in reducing radar detectability, this composite material had significant promise for stealth drone applications. The findings of this study contributed to the advancement of radar-absorbing materials and offered a cost-effective solution for improving the stealth performance of military technology.

SUPPORTING INFORMATION

There is no supporting information in this paper. The data that support the findings of this study are available on request from the corresponding author (NAS).

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CONFLICT OF INTEREST

The authors declared that no conflicts of interest regarding the publication of this article.

AUTHOR CONTRIBUTIONS

RP, AEP, AL, RA, FAS performed the experiment and wrote the early manuscript. NAS supervise the experiment, data calculations, and revise the manuscript. YBA, AKHH, and DDA collaborated on writing and revising the manuscript. All authors approved the final version of the manuscript.

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