

Application of Coatings Based on Chitosan and Eucalyptol in the Protection of Steel against Acid Corrosion

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Abstract

Corrosion is a spontaneous process that can be slowed down, though not entirely prevented, through various methods. The most common method for protecting metals against corrosion involves the application of coatings, with nearly three-quarters of metal structures currently benefiting from organic coatings. In addition to providing effective corrosion protection, modern coatings are expected to be environmentally friendly. Presently, there is growing interest in researching coatings based on water-soluble natural polymers containing active chemical compounds, such as corrosion inhibitors. One such polymer is chitosan, which exhibits excellent film-forming ability in acidic aqueous solutions, making it well suited for diverse protective coating applications. This study explores the possibility of applying a chitosan-based coating with the addition of TiO₂ and eucalyptol for the protection of steel against acid corrosion. The results confirmed that the tested chitosan-based coatings must incorporate an inhibitor to have a protective effect on the steel surface, since these coatings show a corrosive effect without an inhibitor. Although coatings based on chitosan and titanium dioxide with the addition of eucalyptol demonstrate protective effects, the duration of these effects is limited. Consequently, this method of protection could be suitable only for temporary protection of steel against acid corrosion.

Keywords

Chitosan, coatings, corrosion, eucalyptol, steel

1 Introduction

Understanding corrosion is crucial for not only identifying the mechanisms of corrosion, but also for devising effective methods to protect materials from corrosion using appropriate substances, and predicting potential corrosion sites. Common methods for safeguarding metals from corrosion include selecting the appropriate materials, application of electrochemical protection, utilizing corrosion inhibitors, and applying various coatings. Protection of materials with coatings is the most well-known corrosion protection procedure. Coatings must exhibit strong adhesion to the surface and impermeability. Coatings act as physical barriers between the material under protection and the medium in which it is located.^{1,2}

Currently, water-based coatings and those with a high content of dry matter, such as powder coatings, are the most developed and widely used due to increasingly stringent legal regulations governing environmental and human health preservation. Corrosion inhibitors, often added to coatings in very small quantities, play a crucial role in retarding the rate of corrosion. Water-based coatings are increasingly popular owing to their lack of easily volatile, toxic, and flammable solvents, and comprise lower molecular weight substances or binders with good water solubility. However, the disadvantage of these coatings is that they do not provide as good corrosion protection compared to conventional coatings based on organic solvents, necessitating further technological development in this area.³

Due to its advantageous properties, steel is one of the most significant construction materials, used in almost every aspect of human life. Therefore, corrosion protection methods for steel are of paramount importance. Chitosan is a biocompatible and biodegradable polymer derived from chitin, the primary structural polysaccharide in invertebrates and low plants, and the second most abundant polymer in nature after cellulose. In the field of polymer-based coatings, chitosan has emerged as a compelling alternative to conventional coating systems, owing to its exceptional adhesion to metal surfaces and ability to form complexes with potential corrosion inhibitors. Furthermore, chitosan is soluble in water and can be obtained from renewable sources. Recent interest in chitosan as a coating for metal substrates has increased, especially in applications involving magnesium and steel alloys in the biomedical field.^{4–6}

Titanium dioxide (TiO₂) is a chemically inert material utilised in various sectors, including pharmaceuticals, biomedicine, antimicrobial agents, and clean energy. Its widespread application is facilitated by its physicochemical, mechanical, and photocatalytic properties, as well as its thermal stability, low cost, and biocompatibility. Furthermore, the addition of TiO₂ powder into chitosan-based films and coatings has been shown to enhance their mechanical properties.⁷

This study explores the potential application of a coating based on chitosan with the addition of TiO₂ and eucalyptol as inhibitor, against acidic corrosion of steel. The apparent viscosity of the prepared coatings was determined, along with the surface roughness of the steel alloy samples, both with and without coatings. The corrosion rate of the steel samples in acidic solutions was determined gravimetrically,

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while their surfaces were examined using an optical metallographic microscope.

2 Experimental

2.1 Materials

The tests were conducted on alloy steel C. 4230 samples with a diameter of 19 mm and an average thickness of 5 mm. The chemical composition (wt.%) of the alloy steel was as follows: C (0.68–0.77), Si (0.90–1.10), Mn (0.40–0.60), P (max. 0.025), S (0.01–0.03), Cr (0.60–0.80), and balanced Fe. Given that the treatment and cleaning of the metal surface affect the final properties of the coating, the coatings were applied to the steel samples that were either only brushed (using sandpaper grit 400) or polished using SiC paper grit 1200 and diamond paste to obtain a 0.25 μm flat and smooth surface with a mirror-like finish. After processing, all samples were degreased in 96 % ethanol and dried.

2.2 Preparation of chitosan-based solutions and coatings

The chitosan-based solution was prepared by dissolving a specific amount of chitosan (low molecular weight, Sigma-Aldrich, Darmstadt, Germany) in 0.05 % (v/v) acetic acid (80 %, p.a., Gram-mol, Zagreb, Croatia) to achieve a 2 % (w/v) solution. Constant mixing of the solution was conducted for 24 h on a magnetic stirrer. After mixing in the chitosan-based solution, a NaOH solution (1 M) was added to raise the pH value to approximately 6.5, along with 2 % (v/v) glycerol as a plasticizer and 25 % (v/v) 96 % ethanol. Glycerol and ethanol were purchased from Gram-mol (Zagreb, Croatia). The prepared chitosan solution served as the base coating, to which TiO_2 and eucalyptol were added. After stirring for an additional 3 h, the TiO_2 powder was added to the chitosan-based solution (C1) to achieve 5 % (w/v) and 8 % (w/v) TiO_2 solutions. Finally, in each prepared coating, a 30 % (v/v) ethanol solution of eucalyptol was added in a concentration of 1 $\mu\text{l ml}^{-1}$. TiO_2 and eucalyptol were purchased from Sigma-Aldrich, Germany.

Table 1 – Prepared chitosan-based coatings
Tablica 1 – Pripremljeni premazi na bazi kitozana

Coating Premaz	Composition Sastav
C1	2 % (w/v) chitosan solution (basic chitosan solution)
C2	C1 with addition of eucalyptol (1 $\mu\text{l ml}^{-1}$)
C3	C1 with addition of 5 % TiO_2
C4	C1 with addition of 5 % TiO_2 and eucalyptol (1 $\mu\text{l ml}^{-1}$)
C5	C1 with addition of 8 % TiO_2
C6	C1 with addition of 8 % TiO_2 and eucalyptol (1 $\mu\text{l ml}^{-1}$)

The prepared coatings were applied to the steel samples with a surface area of 2.8 cm^2 by the drop casting method using a micropipette to dispense 100 μl of the tested coating onto the surface of each sample. Subsequently, the coatings were dried for 24 h at room temperature before further analysis.

2.3 Methods

2.3.1 Viscosity measurements

The apparent viscosity of all prepared coatings was determined using a rotational viscometer First Plus LR, Lamy Rheology Instruments. The temperature was maintained constant at 25.0 ± 0.2 $^\circ\text{C}$ using a circulating water bath. Viscosity measurements were conducted with the rotational speed of the viscometer measuring cylinder set at 150 rpm. Measurements were taken at 30 measuring points with a duration of 25 s.

2.3.2 Coating thickness determination

The coating thickness was determined based on the difference in steel sample thickness before and after the application and drying of the tested coating using a digital micrometer Insize, 3109-50A. Measurements were taken at several points across each steel sample (min. 8).

2.3.3 Corrosion rate determination

The corrosion rates of the steel samples, both with and without coatings, were determined gravimetrically using an accelerated corrosion test. Accelerated corrosion tests were conducted by exposing the steel alloy samples to acid vapours (10 % HCl) in a closed glass vessel heated to 50 $^\circ\text{C}$ for 2 h. The samples put into the closed vessel were in the presence of an aqueous HCl solution, but not in direct contact with it.⁸

The corrosion rate, W , was calculated from Eq. (1).

$$W = \frac{\Delta m}{S \cdot t} \quad (1)$$

Δm is average weight loss of steel sample (mg), S is the area of the samples in contact with aggressive medium (cm^2), and t is contact time (h).

2.3.4 Surface roughness determination

The surface roughness of the steel samples was tested using a MITUTOYO SURFTTEST SJ-210 portable surface roughness measuring device. Measurements were taken on both bare and coated steel alloy samples after exposure of the samples to hydrochloric acid vapours. To ensure precision, measurements were taken at 15 points on each test sample. Before surface roughness determination, all coatings were removed from the steel surface using tissue paper soaked in water or ethanol.

Table 2 – Values of apparent viscosity and thickness of the prepared chitosan-based coatings (mean \pm SD)Tablica 2 – Vrijednosti prividne viskoznosti i debljine pripremljenih premaza na bazi kitozana (srednja vrijednost \pm SD)

Coatings Prevlake	C1	C2	C3	C4	C5	C6
Viscosity, mPas Viskoznost, mPas	380.64 \pm 0.45	388.24 \pm 0.32	648.67 \pm 0.21	671.47 \pm 0.67	1015.0 \pm 0.45	1032.33 \pm 0.58
Coating thickness, mm Debljina premaza, mm	0.125 \pm 0.006	0.128 \pm 0.004	0.182 \pm 0.008	0.184 \pm 0.005	0.253 \pm 0.010	0.258 \pm 0.012

2.3.5 Optical metallographic analysis

The surfaces of all the steel alloy samples (bare and coated, before and after HCl treatment) were recorded with a metallographic optical microscope Olympus Tokio, Epityp II, Type MO21 at a magnification of 250 \times .

3 Results and discussion

3.1 Apparent viscosity measurements

Viscosity measurements of the prepared chitosan-based solutions were conducted, as viscosity is an important property of coatings and protective materials. Various parameters crucial for coating application are contingent upon viscosity, including material stability during storage, resistance to spillage or leakage from the surface after application, and suitability of the material for specific application procedures. Notably, viscosity is the primary factor influencing coating thickness and adhesion to the substrate.⁹

The apparent viscosity measurement results are expressed as the mean value of 30 measurements, as shown in Table 2.

Analysis of the data reveals a significant increase in the values of apparent viscosity with the addition and increasing content of TiO₂, from 5 % (C3) to 8 % (C5), within the prepared coatings. Furthermore, the addition of eucalyptol as a green corrosion inhibitor to the coating (C2, C4, and C6), led to an increase in viscosity, although not as pronounced as observed in the base coating (C1). It is known that chitosan solutions exhibit pseudoplastic non-Newtonian behaviour, especially at concentrations lower than 2 % (w/v), meaning that their viscosity decreases as the shear rate increases, primarily due to gradual microstructure breakage.¹⁰

Table 2 indicates the expected influence of TiO₂ addition on coating thickness. The thickness of the base chitosan coating (C1) increased from 0.125 mm to 0.182 mm (C3) with the addition of 5 % TiO₂ powder. The thickest coating (C6), achieved with the addition of 8 % TiO₂, measured 0.258 mm.

3.2. Visual observation of the prepared coatings and corrosion rate measurements

Fig. 1 shows the appearance of the polished steel samples after application of the tested coatings using the drop casting method, followed by 24 h of drying at room temperature.

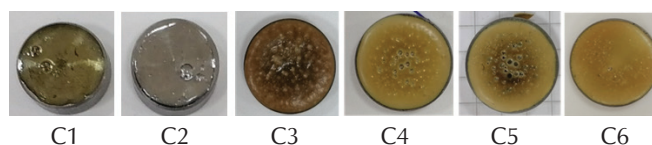


Fig. 1 – Photographs of the polished steel samples after application and drying of the prepared chitosan coatings

Slika 1 – Fotografije uzoraka poliranog čelika nakon nanošenja i sušenja kitozanskih premaza

It is evident that the prepared coatings lacking an added inhibitor (C1, C3, and C5) induced changes on the steel surface, suggesting a reaction between the metal surface and the coating. Also noteworthy is the significant colour change observed in the coating consisting of chitosan and 5 % TiO₂ without added inhibitor (C3) compared to the other prepared coatings. Based on this observation, it can be assumed that a reaction occurred between the steel surface and the applied coating, potentially indicating the onset of corrosion beneath this coating. A similar effect was also observed with coating C5 (containing 8 % TiO₂ and no inhibitor). Additionally, the surfaces obtained after drying coatings C3–C5 exhibit an inhomogeneous structure, whereas only coatings C2 and C6 displayed a relatively homogeneous structure. The addition of the inhibitor resulted in a less pronounced colour change (C2, C4, and C6), indicating that the reaction between the coating and the steel surface was inhibited. The same observations were made with the steel samples that were only brushed.

Since chitosan-based coatings are easily removed from the steel surface, conventional electrochemical measurements involving immersion in water solutions cannot be employed to quantify their protective efficacy, as they would lead to coating dissolution. Therefore, the protective efficacy of the coatings was investigated through accelerated corrosion treatments in the presence of aggressive species capable of promoting degradation processes and providing rapid information. The polished steel samples, with and without the tested coatings, were exposed to 10 % hydro-

chloric acid vapours for 2 h at 50 °C.⁴ Based on the difference in mass before and after exposure to hydrochloric acid vapours, the corrosion rate values were calculated. The calculated corrosion rate values for the polished and brushed steel samples, expressed in $\text{mg cm}^{-2} \text{h}^{-1}$, are presented in Fig. 2.

Fig. 2 illustrates that the steel samples with coatings without eucalyptol as inhibitor (C1, C3, and C5), exhibited higher corrosion rates compared to the bare steel, indicating that coatings without inhibitors do not offer protective benefits to polished and brushed steel. This result is correlated with the visible colour change on the steel surfaces with coatings C1, C3, and C5 after drying. The addition of eucalyptol to coatings C2, C4, and C6 resulted in a decrease in the corrosion rate values, although not significantly compared to the effect of hydrochloric acid on the bare steel samples. These results also indicate that the addition of TiO_2 to the base chitosan coating without a corrosion inhibitor leads to significant steel corrosion.

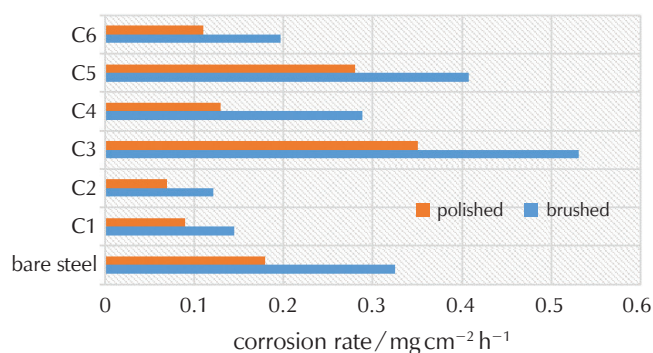


Fig. 2 – Corrosion rates ($\text{mg cm}^{-2} \text{h}^{-1}$) of steel samples after accelerated corrosion test

Slika 2 – Brzine korozije ($\text{mg cm}^{-2} \text{h}^{-1}$) uzoraka čelika nakon ubrzanog korozijskog ispitivanja

From Fig. 2 it is also evident that the corrosion rate was significantly higher on the samples that were only brushed compared to those that were polished. This behaviour was expected, as higher surface roughness leads to stronger corrosion. It is important to note that despite the identical dimensions of all steel samples, the surfaces of the brushed samples were larger due to their roughness compared to the same polished steel samples, potentially leading to deviations in the obtained results from the actual values.

Fig. 3 shows the surface appearance of polished steel samples after the accelerated corrosion test. Although the coatings were partially retained on the steel samples after the accelerated corrosion test, they were removed to expose the steel surface for optical metallography imaging. Various effects of the prepared chitosan-based coatings on the metal surface are clearly discernible. The surfaces of the steel samples that were coated without inhibitors (C1, C3, and C5) were significantly more damaged, the changes being readily apparent upon observation. The least damage, or surface changes, were visible in samples C2, C4, and C6,

the coatings of which contained eucalyptol as an inhibitor.

Similar behaviour was observed on the samples of only brushed steel surfaces.

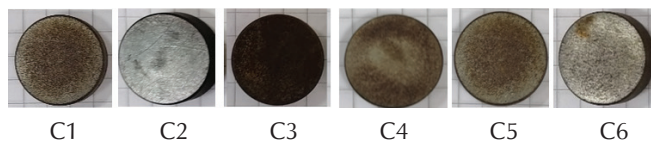


Fig. 3 – Photographs of polished steel samples after the accelerated corrosion test

Slika 3 – Fotografije uzoraka poliranog čelika nakon ubrzanog korozijskog ispitivanja

3.3. Surface roughness determination

Surface roughness measurements were conducted on both polished and brushed steel samples, before and after exposure to an aggressive acidic medium. For this purpose, the coatings were removed from the steel surface allowing for the determination of the surface roughness value of the pure metal. The results are presented as the vertical roughness parameter, R_a , which represents the mean arithmetic deviation of the profile. In other words, on the unit length of the surface, the mean value is calculated from the total values of the roughness amplitudes. For the measurements, a probe was employed to pass over the surface of the samples and measure deviations at arbitrarily selected points.¹¹

Table 3 presents the values for the vertical roughness parameter, R_a , measured on the steel samples with and without the tested coatings, before and after exposure to HCl vapours. Before surface roughness determination, all coatings were removed from the steel surfaces using tissue paper soaked in water or ethanol. The results clearly indicate that the R_a parameter values significantly increased after exposing the steel surface to an aggressive acidic medium, signifying an advanced corrosion process.

Furthermore, it was evident that coatings lacking eucalyptol as a corrosion inhibitor (C1, C3, and C5) on both the polished and brushed steel samples, after application and drying for 24 h, led to a slight increase in surface roughness, indicating an obvious reaction of these coatings with the steel surface. These results suggest that the coatings prepared without an inhibitor contained aggressive ions that led to steel corrosion. Despite raising the pH value of the base chitosan solution to 6.5 by adding NaOH solution, it was evident that the coatings contained both acetate and hydrogen ions, known to induce corrosion on steel surfaces.¹² The steel samples with the C1, C2, and C6 coatings exhibited the least impact on surface roughness, proving that these coatings did not have a corrosive effect on the steel. The highest values of the R_a parameter were obtained with the C3 coating, containing 5 % TiO_2 without eucalyptol, while the lowest values were observed with the coating containing only chitosan but with the addition of eucalyptol.

Table 3 – Values of the vertical surface roughness parameter, R_a , on the tested steel samples before and after exposure to acidic medium (mean \pm SD)

Tablica 3 – Vrijednosti parametra vertikalne hrapavosti površine, R_a , na ispitivanim uzorcima čelika prije i nakon izlaganja kiselim mediju (srednja vrijednost \pm SD)

Steel samples Uzorci čelika	Before exposure, $R_a/\mu\text{m}$ Prije izlaganja, $R_a/\mu\text{m}$		After exposure, $R_a/\mu\text{m}$ Nakon izlaganja $R_a/\mu\text{m}$	
	Polished Polirani	Brushed Brušeni	Polished Polirani	Brushed Brušeni
Without coating	0.37 ± 0.03	0.88 ± 0.06	1.35 ± 0.08	3.45 ± 0.21
Coating 1	0.55 ± 0.02	1.23 ± 0.08	1.17 ± 0.06	2.24 ± 0.04
Coating 2	0.50 ± 0.04	1.06 ± 0.05	1.00 ± 0.11	1.90 ± 0.04
Coating 3	1.51 ± 0.05	1.85 ± 0.12	3.54 ± 0.23	4.01 ± 0.15
Coating 4	1.35 ± 0.08	1.80 ± 0.09	2.83 ± 0.17	3.66 ± 0.22
Coating 5	1.24 ± 0.06	1.42 ± 0.11	3.01 ± 0.14	3.23 ± 0.13
Coating 6	0.87 ± 0.03	1.28 ± 0.08	1.53 ± 0.08	2.46 ± 0.15

3.4 Optical metallographic analysis

The surfaces of the steel samples were analysed using an optical metallographic microscope at a magnification of $250\times$ to gain insight into the condition of the steel surface before coating application and after the coatings were dried for 24 h at room temperature. The coatings were removed from the surface of the steel for this analysis, and the steel surfaces were recorded after both uncoated and coated samples were exposed to aggressive hydrochloric acid vapours.

Fig. 4 shows the surfaces of the polished steel before exposure to the acidic medium without coating, and the surfaces of the steel after C2 and C6 coatings were applied to the steel. It is evident from the image that hydrochloric acid does not cause significant damage to the surface of the polished steel, whereas the damage is far more extensive on the steel that had only been brushed (Fig. 5). It

is also evident that hydrochloric acid caused considerable damage to the surface of the brushed steel, manifested as tiny holes, confirming the occurrence of pitting corrosion on steel upon exposure to chloride ions.^{13,14} These results clearly suggest that surface preparation profoundly impacts the final outcome and the rate of the corrosion process itself. Despite visible damage on the steel surface, even after exposure to an aggressive acidic medium, the damages were significantly smaller on samples protected by the C6 coating compared to those on the unprotected steel surfaces, especially on the brushed steel.

4 Conclusion

Water-based coatings containing renewable and readily available polymeric materials such as chitosan present a promising alternative to existing organic-based coatings.

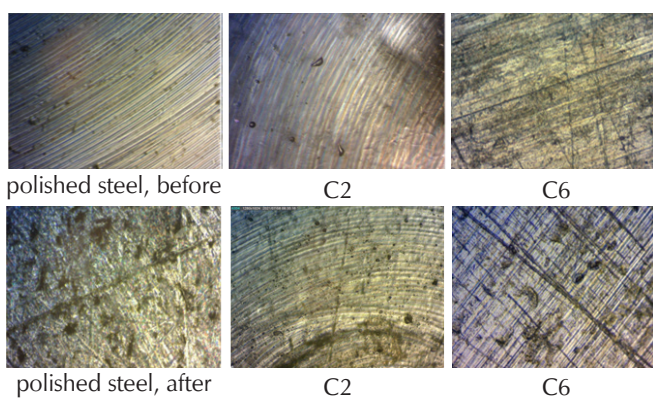


Fig. 4 – Polished steel surface without coating, and with coatings C2 and C6, before and after exposure to acidic media recorded with an optical metallographic microscope

Slika 4 – Površine poliranog čelika bez premaza i s premazima C2 i C6 prije i nakon izlaganja kiselim mediju snimljene optičkim metalografskim mikroskopom

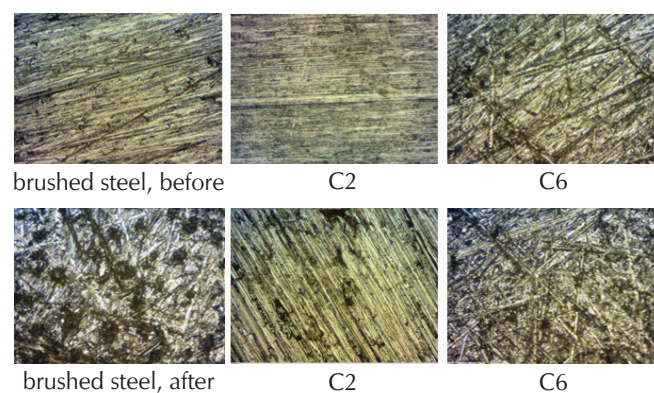


Fig. 5 – Brushed steel surface without coating, and with coatings C2 and C6, before and after exposure to acidic media recorded with an optical metallographic microscope

Slika 5 – Površine brušenog čelika bez premaza i s premazima C2 i C6 prije i nakon izlaganja kiselim mediju snimljene optičkim metalografskim mikroskopom

These coatings, consisting of environmentally friendly polymer materials, not only act as barrier layers, but can also encapsulate corrosion inhibitors. This study demonstrates that chitosan can serve as a polymer matrix for water-based coatings. However, it is crucial for such coatings to incorporate an inhibitor as otherwise they may not prevent corrosion on steel surfaces. The chitosan coating with eucalyptol, C2, and coating C6, with 8 % TiO₂ and eucalyptol, exhibited superior surface appearance, and demonstrated the best protective properties and resistance against the action of aggressive hydrochloric acid. While coatings based on chitosan and titanium dioxide with the addition of eucalyptol showed protective effects, it should be noted that these coatings are water-soluble and that the duration of their protective effects is limited. Therefore, this method of protection could be suitable only for temporary protection of steel against acid corrosion.

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List of abbreviations and symbols

Popis kratica i simbola

- C1 – basic chitosan solution
 C2 – basic chitosan solution with addition of eucalyptol, 1 μl ml⁻¹
 C3 – basic chitosan solution with addition of 5 % TiO₂
 C4 – basic chitosan solution with addition of 5 % TiO₂ and eucalyptol, 1 μl ml⁻¹
 C5 – basic chitosan solution with addition of 8 % TiO₂
 C6 – basic chitosan solution with addition of 8 % TiO₂ and eucalyptol, 1 μl ml⁻¹
 R_a – vertical roughness parameter, μm
 SD – standard deviation
 W – corrosion rate, mg cm⁻² h⁻¹

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SAŽETAK

Primjena premaza na bazi kitozana i eukaliptola u zaštiti čelika od kiselinske korozije

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Korozija je spontani proces koji se ne može u potpunosti spriječiti, ali se može usporiti primjenom različitih metoda. Najčešći način zaštite metala od korozije je nanošenje premaza, pa se danas gotovo 3/4 metalnih konstrukcija štiti organskim premazima. Osim učinkovite zaštite od korozije, od modernih premaza očekuje se i ekološka prihvatljivost. Danas se sve više istražuju premazi na bazi prirodnih polimera koji su topljivi u vodi, a sadrže i aktivne kemijske spojeve poput inhibitora korozije. Jedan od takvih polimera je kitozan koji u kiselim vodenim otopinama ima izvrsnu sposobnost stvaranja filma, pa je pogodan za primjenu u raznim područjima zaštitnih premaza. U ovom radu ispitana je mogućnost primjene premaza na bazi kitozana s dodatkom TiO₂ i eukaliptola kao zaštite čelika od kiselinske korozije. Potvrđeno je da ispitani premazi na bazi kitozana moraju sadržavati inhibitor da bi imali zaštitno djelovanje na površini čelika, jer bez inhibitora ti premazi pokazuju korozivno djelovanje. Iako premazi na bazi kitozana i titanijeva dioksida s dodatkom eukaliptola pokazuju zaštitni učinak, njihov zaštitni učinak ne može biti dugotrajan, pa se taj način zaštite može primjenjivati samo kao privremena zaštita čelika od kiselinske korozije.

Ključne riječi

Čelik, eukaliptol, kitozan, korozija, premazi

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