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GRAVIMETRIC METHOD OF CORROSION PROTECTION FOR STEEL N20 IN AN ACIDIC ENVIRONMENT

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Abstract

In this study, carbon dots were synthesized using chitosan and thiourea via hydrothermal treatment at 180 °C for 4 hours. The synthesized carbon dots were characterized by Transmission Electron Microscopy (TEM) analysis. The size of the synthesized carbon dots was found to be in the range of 8 nm to 10 nm. These carbon dots were used as a green corrosion inhibitor to mitigate the corrosion of mild steel (MS) in 0.5 M HCl solution using the gravimetric method. The studied carbon dots exhibited an inhibition efficiency of 91.70% at a concentration of 200 mg/L and a temperature of 303 K. The observed corrosion inhibition is attributed to the adsorption of the carbon dots onto the MS surface.

Keywords: *corrosion inhibitors, metal surface, carbon dots, solution, concentrations*

1. Introduction

Corrosion of metallic materials, particularly carbon steel (KP), remains a significant challenge in industrial operations such as oil and gas extraction, chemical processing, acidizing, and desalination systems. This leads to substantial economic losses not only due to the replacement of damaged infrastructure but also because of maintenance, downtime, and reduced equipment efficiency (He, H., et al., 2025; Yan, H., et al., 2024). Beyond the economic burden, corrosion also gives rise to serious environmental and safety hazards, including the leakage of toxic chemicals, equipment failure, and catastrophic accidents. Therefore, the need for reliable, efficient, and environmentally benign corrosion control methods is urgent and widely recognized. Among the numerous approaches to corrosion mitigation, the addition of inhibitors to corrosive environments is one of the most cost-effective strategies. Inhibitors significantly reduce the corrosion rate by forming a protective layer on the metal surface and preventing the penetration of corrosive agents.

Corrosion inhibitors can be inorganic or organic in nature (Iravani, D., et al., 2024). Common inorganic inhibitors such as chromates, phosphates, and nitrites are increasingly being phased out due to their toxicity, environmental persistence, and harmful effects on human health. Organic corrosion inhibitors have attracted interest due to their excellent corrosion control efficiency and ease of application. However, the hazardous nature and relatively high cost of many effective organic inhibitors have driven the search for cost-effective, environmentally safe, and sustainable alternatives. Recently, nanotechnology has opened new possibilities in the field of corrosion control through the use of nanomaterials. Nanostructured materials, owing to their high surface-to-volume ratio and versatile surface chemistry, exhibit strong interactions with metallic substrates. Carbon dots (CDs) are a particularly interesting class of nanomaterials for next-generation green corrosion inhibitors (Tang, M., et al., 2026). They are cost-effective, environmentally acceptable, and easy to prepare as zero-dimensional carbon nanoparticles with sizes below 10 nm. Fluorescence is an inherent property of CDs. Synthesizing CDs from biowaste is advantageous because it supports waste management and the production of carbon-based compounds (Tang, Y., et al., 2024). The excellent water dispersibility, low toxicity, chemical inertness, environmental friendliness, and tunable luminescence of CDs have attracted significant attention since their discovery in 2004, leading to their widespread application in biomedicine, sensing, catalysis, lubrication, and other fields.

Figure 1. *Synthesis process*

An additional advantage of using biomass precursors for CD synthesis is their ability to promote endogenous heteroatom doping. Natural biomass sources contain heteroatoms such as oxygen, nitrogen, sulfur, and phosphorus within their molecular frameworks (Wang, Q., et al., 2024; Islami, L.A. and Anawati A., 2025; He, C., et al., 2024). During hydrothermal or pyrolytic carbonization, these heteroatoms can spontaneously incorporate into the carbon dot structure or surface functional groups without relying solely on external dopant reagents (Lv, Y., et al., 2023; Ren, S., et al., 2022). This endogenous doping results in a high degree of surface functionalization, which is particularly beneficial for corrosion inhibition as it enhances interfacial adsorption, electron donor capability, and film-forming ability on metallic substrates.

This article reviews the synthesis and anti-corrosion inhibitory performance of carbon dots derived from chitosan as inhibitors.

2. Materials and methods

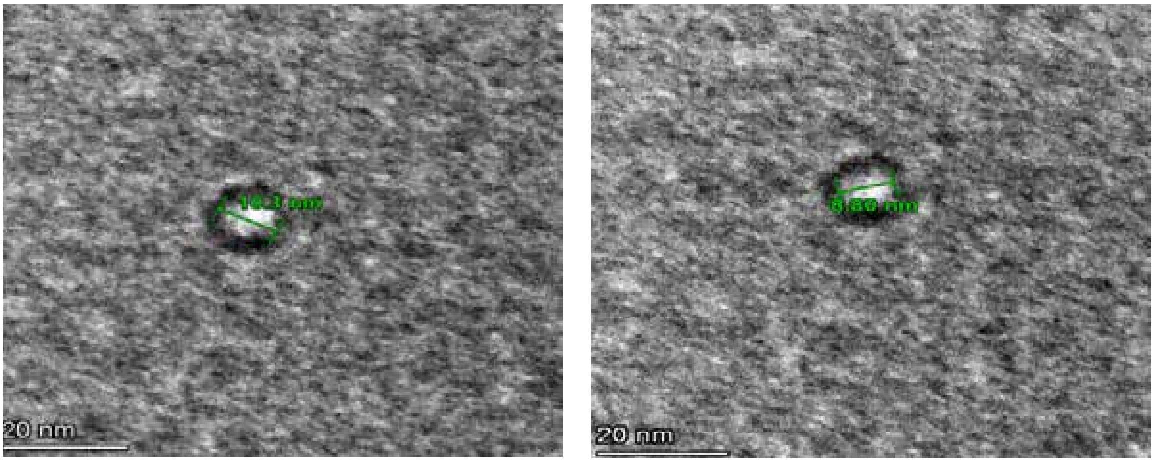
2.1. Materials: Chitosan and thiourea reagents were supplied by Shaanxi Alibon Biotechnology Co., Ltd. Hydrochloric acid (33%) was obtained from Henan Refortune Industrial Co., Ltd.

2.2. Preparation: Chitosan and thiourea were weighed in equal proportions (250 mg each) on an analytical balance and dissolved in 100 mL of distilled water. This mixture was stirred using an ultrasonic mixer. The solution was placed in an autoclave and subjected to a hydrothermal reaction in a muffle furnace at 200 °C for 4 hours. The obtained carbon dots were purified in several steps, for example, filtration, dialysis, and centrifugation at 12,000 rpm at 10 °C for 15 minutes, yielding pure carbon dots. The resulting carbon dots were dried at 40 °C to obtain dry carbon dots. The synthesis reaction of the new carbon dots based on chitosan and thiourea can be represented as follows (figure 1).

3. Experimental results and discussion

3.1. Transmission electron microscope (TEM) analysisThe size of the synthesized chitosan- and thiourea-based carbon dots was determined by TEM to be in the range of 8–10 nm, and their morphology was found to be spherical in shape. This indicates that the small-sized carbon dots were successfully synthesized (figure 2).

Figure 2. *Transmission electron microscope*



3.2. Gravimetric analysis: The anti-corrosion inhibition properties of the obtained carbon dots were examined using the gravimetric method. Gravimetry is a widely used method in analytical chemistry that is based on mass measurement for determining substances and their quantities. In the gravimetric method, metal plates of various N20 steel samples with dimensions of 3.0 × 3.0 × × 0.2 cm were used. Before starting the experiments, the metal samples were cleaned with emery paper and the metal dimensions were determined. They were then washed with distilled water and acetone. The experiments were carried out in a 0.5 M HCl solution at various time intervals. Initially, the metal sample was immersed in the test solution for 3 hours, after which its mass was weighed. Then, various concentrations of inhibitors were added, and the metal sample was weighed again. Before weighing, the metal samples were washed in distilled water, then in acetone, and subsequently weighed on a balance. Each experiment was repeated three times. The obtained results were recorded(1) (de Souza, F. S. and Spinelli A., 2009).

$$\eta = \frac{w_o - w_{ing}}{w_o} \times 100\% \qquad (1)$$

where *wo* and *wing* are the weight losses (mg) in the absence and presence of the inhibitor, respectively, and η% is the inhibitor efficiency. The surface coverage degree (θ) was found using the following formula(2):

$$\theta = \frac{w_o - w_i}{w_o} \qquad (2)$$

The corrosion rate was found using the following formula(3):

$$K'' = \frac{8.76\times10^{4}W}{D\times A\times t} \quad (3)$$

where W is the weight loss (mg), A is the surface area of the sample immersed in the test solution (cm^2), t is time (hours), and D is the density of the metal sample (g/cm^3). Conducting experiments at different temperature ranges and various inhibitor concentrations when determining the efficiency of inhibitors using the gravimetric method, along with its high degree of accuracy, indicates that the essence of this method is substantial. At the same time, it should be noted that the significant amount of time required to determine the inhibitor efficiency is a disadvantage of this method. In order to determine the optimal concentration of the research objects in the background solutions, studies were carried out at various concentrations of the inhibitors.

Table 1. *Results of determining the efficiency of new carbon dots based on chitosan and thiourea as inhibitors at various temperatures and concentrations*

C_{ing}, mg/l	Temperature, K	$CR_{Grav.}$, mg/cm² h	$\eta_{Grav.}$, %	$\theta_{Grav.}$
HCl		1,236	–	–
50		0,2948	76,15	0,7615
100	303	0,2365	80,87	0,8087
150		0,1899	84,64	0,8464
200		0,1026	91,70	0,9170
HCl		1,584	–	–
50		0,4567	71,17	0,7117
100	313	0,3568	77,47	0,7747
150		0,2758	82,59	0,8259
200		0,1989	87,44	0,8744
HCl		1,759	–	–
50		0,5896	66,48	0,6648
100	323	0,4256	75,80	0,7580
150		0,3501	80,10	0,8010
200		0,2985	83,03	0,8303
HCl		1,847	–	–
50		0,6855	62,89	0,6289
100	333	0,4985	73,01	0,7301
150		0,4134	77,62	0,7762
200		0,3869	79,05	0,7905

To obtain more information about the surface coverage of the steel by the inhibitor and the protection mechanisms, the temperature dependence of the degree of protection of the chitosan- and thiourea-based inhibitor was studied (Table 1). Studies were conducted using the gravimetric method at various concentrations while varying the temperature from 303 K to 333 K in increments of 10 °C. The increase in the degree of protection with increasing temperature indicates that the inhibitor is adsorbed onto the steel surface as a result of chemical adsorption. A lesser increase in the degree of protection with increasing temperature indicates that physical adsorption is occurring alongside chemical adsorption in the process. The simultaneous occurrence of both chemical and physical adsorption indicates that the inhibitor is a mixed-type inhibitor. At 303 K, a maximum degree of protection of 91.70% was achieved for the chitosan- and thiourea-based inhibitor at a concentration of 200 mg/L.

Conclusion:

Chitosan- and thiourea-based carbon dots were synthesized, and the optimal conditions for their synthesis were established. The anti-corrosion inhibition properties of the obtained carbon dots were also determined via the gravimetric method. In this case, gravimetric studies were carried out at various temperatures and various concentrations. The best result showed a degree of protection of 91.70% at 30 °C and 200 mg/L. In conclusion, it can be said that the obtained carbon dots are readily soluble in water, biocompatible, non-toxic, and exhibit good inhibition properties.

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