



# Enhancement of the wear resistance of epoxy coating in presence of MBT-loaded mesoporous silica nanocontainers



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## ABSTRACT

The impact mechanism of mesoporous 2-Mercaptobenzothiazole (MBT)-loaded silica nanocontainers (SNCs) on wear resistance of epoxy coating was investigated. Scanning electron microscopy, BET analysis, UV-Vis spectrophotometry and thermogravimetric analysis were used to characterize SNCs. Also, pin-on-disk wear test, microhardness measurement and electron microscopy were used to investigate the impact mechanism of MBT-loaded SNCs on wear resistance of epoxy coating. Results of wear analysis revealed that presence of MBT-loaded SNCs increased the coating microhardness and also decreased the friction coefficient of coating layer and thereby resulted in significant improvement of wear resistance of coating. It was concluded that the superior wear resistance of nanocomposite coatings in presence of silica nanocontainers is due to reinforcement effect and self-lubricating action of mesoporous MBT-loaded SNCs.

## 1. Introduction

Protective coatings and primers have been used extensively to protect the metallic substrates against corrosion. In a conventional organic coating, the inhibitor species are added to actively protect the substrate against corrosion while the coating matrix provides a physical barrier against aggressive species [1,2]. Although the use of inhibitor-loaded organic coatings can provide active corrosion protection, the direct addition of inhibitor molecules into the coating layer suffer from several drawbacks such as undesirable interaction between inhibitor molecules and coating matrix and also uncontrolled leach out of the corrosion species which leads to loss of inhibition efficiency within a relatively short period of time [3].

The need to overcome the abovementioned drawbacks has guided the attentions toward development of smart protective coatings which provide on-demand release of corrosion inhibitor. According to this strategy, the inhibitor species would be encapsulated or intercalated into nanocontainers and then dispersed in the coating formulation [4–12]. The subsequent release of inhibitor species would be controlled by various environmental triggers such as ionic strength, change of pH, the presence of aggressive species and also mechanical impact loadings to cracks in the coating [1,4,13,14]. A considerable amount of works related to the corrosion protection mechanism of these coatings have been done by several authors [3], but the investigations involving the tribology of

these coatings are still limited. However, since protective coatings are used in a wide variety of industrial applications which are subjected to mechanical damages such as sliding and abrasive wear, the reinforcement of coating layer can improve the mechanical properties of the coating against environmental damages and enhance the effectiveness of coating matrix to provide a promising physical barrier against aggressive species. Several works have been done to investigate the effect of various nanoparticles on the tribological behavior of epoxy composites. It has been reported that the addition of a rather low weight fraction of Ni particles would reduce the coefficient of friction and the specific wear rate of epoxy resin [15]. Also, Kurahatti et al. have reported that the specific wear rate of an epoxy composite with 0.5 wt % of ZrO<sub>2</sub> nanoparticles is decreased by 95% as compared to the value of unfilled epoxy [16]. In another work, it has been shown that the addition of a high amount (25%) of nanosilica would enhance the wear properties of epoxy polymer [17]. Also, McCook et al. studied the tribological properties of epoxy coating containing various volumetric percentages of PTFE and ZnO. They have reported that tribological properties of the epoxy nanocomposites can be greatly influenced with the addition of small volume percent of zinc oxide and PTFE nanoparticles [18].

Recently, we have developed an epoxy coating containing 2-Mercaptobenzothiazole (MBT)-loaded silica nanocontainers (SNC) to provide active inhibition action for corrosion protection of carbon steel substrate [3]. According to the results of our study, incorporation of MBT-loaded

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SNCs resulted in potent corrosion protection performance of the epoxy coating. However, since SNCs are hard ceramic particles, it is expected that incorporation of MBT-loaded SNCs would result in reinforcement of epoxy matrix. According to literature, incorporation of silica nanoparticles into epoxy coating would result in enhancement of tribological properties of the coating matrix [19,20]. In this regard, it has been reported that in comparison with micrometer silica, the presence of nanosilica can result in better wear properties due to the simultaneous decrease of friction and wear functions of the epoxy matrix [21]. Also, it has been shown that the addition of mesoporous silica particles leads to lower friction in epoxy matrix [22].

In addition, since MBT is an organic molecule, the encapsulation of MBT species into mesoporous SNCs can result in self-lubricating action of coating layer against wear in service conditions. To the best of our knowledge, investigation of the wear resistance of epoxy coating with MBT-loaded SNCs is a new topic and there has been no report on the influence of MBT-loaded SNCs on the wear resistance of epoxy coating. The aim of present study is to investigate the effect of MBT-loaded SNCs on the wear resistance of epoxy coatings.

## 2. Materials and methods

### 2.1. Materials

Cetyltrimethylammonium bromide (CTAB) (99%), potassium persulfate (KPS), sodium chloride (NaCl), 2-mercaptobenzothiazole (MBT) (97%), polyvinylpyrrolidone (PVP), Tetraethyl orthosilicate (TEOS) (99.9%) and styrene (St) were prepared from Merck Company. Also, ammonia solution (NH<sub>4</sub>OH) (25–28%) was purchased from Arman Sina company and Ethanol (EtOH) (96%) was purchased from Taghtir Khorasan.

### 2.2. Synthesis methods

Mesoporous SNCs were synthesized as described elsewhere [3]. In a typical experiment, ethanol and distilled deionized water with the volume ratio of 150:255 were mixed together to form a solution. In the next step, 5 mL of TEOS was gently added to the solution under stirring. The solution was stirred for 10 min and then 80 mg of CTAB surfactant was added to the solution. In the next step, the solution was mixed with 5 mL of NH<sub>4</sub>OH and then was stirred for 3 h at room temperature. Then, in order to collect the mesoporous SNCs, the solution was centrifuged at 10000 rpm for 20 min. Finally, SNCs were thoroughly washed with ethanol and dried at 60 °C for 8 h. In order to assure complete removal of organic impurities, the silica nanocontainers were heat treated at 550 °C for 5 h.

In addition, SNCs with larger diameters were synthesized to investigate the effect of particle size of SNCs. For this purpose, SNCs were synthesized according to Stober procedure as follows [23]. First, 2 g of PVP was mixed with 100 mL of distilled deionized water. The mixture was stirred for 15 min and 10 g of styrene was added to the mixture and the mixture was transferred to an Erlenmeyer flask. In the following, the Erlenmeyer flask was immersed in a hot water bath at 70 °C. In the next step, 0.2 g of KPS was added into the reaction chamber as the initiator until the polymerization process starts. The reaction chamber was kept in hot water for 24 h and then was cooled down to room temperature to give a polystyrene suspension.

In the next step, the suspended polystyrene particles were used as solid templates for fabrication of silica-coated polystyrene particles. Accordingly, 5 g of the prepared polystyrene suspension was added to 120 mL of ethanol and stirred for 20 min. Then, 4 mL of NH<sub>4</sub>OH and 5 mL of TEOS were added to the solution and the mixture was kept at room temperature and stirred for 18 h to prepare the silica coated polystyrene particles. Then, silica-coated polystyrene particles were collected by centrifugation at 8000 rpm for 20 min. In the next step, silica-coated polystyrene particles were washed thoroughly by ethanol and distilled

deionized water and dried at room temperature for 24 h. Finally, in order to prepare empty silica nanospheres, the polystyrene cores were removed by annealing at 500 °C for 6 h.

### 2.3. Preparation and characterization of MBT-loaded silica nanocontainers

For preparation of MBT-loaded SNCs, the mesoporous silica nanocapsules were dispersed in a 20 mg mL<sup>-1</sup> solution of MBT in acetone. The mixture was then mixed for 36 h at room temperature. Then, the MBT-loaded silica nanocapsules were collected by centrifugation at 8000 rpm. In the next step, the MBT-loaded SNCs were washed thoroughly with distilled deionized water and dried at 60 °C for 8 h.

The morphology and particles size of MBT-loaded SNCs were investigated by using a TE-SCAN scanning electron microscope (model MIRA3). Also, in order to investigate the successful loading of SNCs with MBT molecules, the UV-Vis absorption spectrum of the ethanol suspension of empty SNCs was compared with that of MBT-loaded SNCs. The UV-Vis analyses were done by using a Shimadzu UV-Vis spectrophotometer. In addition, the BET analysis was used to characterize the porosity and specific surface area of SNCs. The BET analysis was carried out by using a BEL: belsorp-mini-2 instrument. The BET analysis were conducted based on the adsorption-desorption isotherms of N<sub>2</sub> at -196 °C. Prior to BET analysis, the MBT-loaded SNCs were degassed at 120 °C for 6 h.

Thermogravimetric (TG) analysis was performed to measure the amount of MBT loading in SNCs. TG analysis was conducted by using a Bahr: STA 503 thermal analyzer under air atmosphere. The sample temperature was increased from room temperature to 700 °C at a heating rate of 10 °C min<sup>-1</sup>.

### 2.4. Characterization of wear resistance of coatings

The specimens for wear resistance measurements were rectangular coated samples of mild steel. In order to prepare the specimens, the carbon steel substrates of 2 cm × 2 cm × 0.2 cm dimensions were blast cleaned to a Sa-2.5 grade (Swedish standard). Then, the metallic substrates were coated with various coating compositions by using air spray. A commercially available epoxy primer (provided by Firoozeh Paint and Resin Co) was used to prepare coatings. The paint formulation consisted of an alkyd resin and a polyamide hardener with a weight ratio of 14:1. Various coating compositions with 1, 1.5 and 2 wt% of MBT-loaded SNCs were prepared by addition of the required amounts of MBT-loaded SNCs to epoxy primer. The SNCs were uniformly dispersed into the epoxy resin via ultrasonication for 30 min and then the mixture was further stirred for 1 h at room temperature. In order to apply the coating layers, a stoichiometric amount of hardener was added to the mixture under stirring. Thickness of dry coating layer for all coated samples was measured to be 100 μm.

The wear resistance of coated samples was measured by using a pin-on-disk tribometer. AISI 52100 steel pins with chromium coating (having the diameter of 6 mm and height of 48 mm) has been used as counterpart. The applied normal load and sliding velocity were 210 N and 100 mm s<sup>-1</sup>, respectively. The weight loss of samples after 150 m of dry sliding was measured to evaluate the erosion resistance of coatings. Also, the pin-on-disk tribometer was used to determine the coefficient of friction of each sample after 150 m of dry sliding. The frictional force was measured by the transducer mounted on the loading arm. Each measurement was repeated more than three times and error bars were shown on the corresponding graphs. Also, scanning electron microscopy was used to characterize the surface appearance of coated samples after wear measurements.

Also, microhardness measurements were performed by using a Koopa Vickers microhardness tester.

In addition, a Cambridge scanning electron microscope (model Cambridge-360) was used to observe the surface configuration and

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