



Epoxy/ PVC/ Nanosilica Hybrid Coating as an Organic Coating with Enhanced Thermal Stability and Wear Resistance

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ABSTRACT

Low resistance at high temperatures as well as low wear resistance are the limitations of polymer-based materials; therefore, to increase thermal stability and wear resistance of these materials, resistant coatings used to protect their surface in applications with good wear performance at high temperatures. The coating layer in this study includes epoxy resin mixed with polyvinyl chloride (PVC) as the organic phases, containing 5, 7.5, 10, 12.5, 15 and 17.5 %wt of the modified silica nanoparticles as inorganic phase are applied to polymeric substrates through dip-coating. Wear resistance, hardness and thermal gravimetry analysis (TGA) were used to evaluate the properties of applied hybrid coating. The results showed that the thermal stability of the substrate with Epoxy/PVC coating containing 12.5 %wt modified nanosilica had an increase up to 31.27% compared to the uncoated substrate. The hardness obtained in the uncoated substrate is 2H; While in substrates with coatings containing 12.5 and 15% wt of modified nanosilica increased to 6H. The wear rate of substrate with Epoxy/PVC hybrid coating containing 12.5 %wt of modified nanosilica had amount 75% reduce compared to the uncoated substrate. The results showed the potential ability of the formulated coating to enhance the working conditions of polymeric coating including high temperature and high wear conditions.

Keywords: Wear resistance; Thermal stability; Nanosilica; Hybrid coating

1. Introduction

The use of polymer composites has received special attention in recent years. These materials are widely used in the automotive, aerospace, construction and sports equipment industries. For example, the use of these materials in Boeing 787 and Airbus 380 aircraft has increased to more than 50%. The reason for the increasing use of these composites is due to the special advantage of these materials. Their most important characteristics are high strength to weight ratio, good fatigue resistance, and good corrosion resistance. However, thermal and wear resistance at high temperatures and times are main weaknesses of these composites [1, 2]. In these composites, increasing the temperature

of the substrates in different working conditions leads to their combustion and destruction. This degradation is greater for substrates with low operating temperatures and therefore is a major challenge in the application of these materials. Therefore, many researches are ongoing to enhance the thermal and wear resistance of these organic coatings for use in various industries [3, 4].

Various coatings used for protection of polymeric composites against wear and temperature at high operating temperatures and times, including ceramic, metal, and thermal barrier coatings; which is applied by thermal spraying and HVOF (High Velocity Oxygen Fuel) methods, which are considered very expensive. Moreover, in using these

coating methods, since the operating temperature is very high, many parameters must be controlled [5, 6].

Composite coatings containing various nanoparticles that usually applied by dipping method can be desirable alternatives for expensive methods. By improving the surface properties, these coatings help to increase the useful life of manufactured parts, reduce maintenance costs and increase the operating range of polymer-based composites [7, 8]. One of these types of coatings is epoxy/nanosilica based composite coatings that used to expand the application of these coatings in various industries [9]. One of the most important advantages of these coatings is the formation of chemical bonds with the substrate surface and reinforcing particles, which occurs due to the reaction between molecular precursors and hydroxyl functional groups, resulting in very strong adhesion. The variety of raw materials and achieving the desired properties by modifying the chemical surface or adding some reinforcing particles to these coatings has led to the development of their applications in various fields both at low temperatures and at high temperatures [9, 10].

Van et al. [11], developed epoxy/silica wear-resistant coatings by modifying the surface of silica powder and its adding in a certain ratio to epoxy resin. The results of this study showed that adding a certain amount of surface-modified silica powder, curing agent and additive to epoxy resin leads to wear-resistant coating. The results reported by Nguyen et al [12] on the study of the effect of nanoparticles on the mechanical and thermal properties of epoxy coatings showed that the addition of titanium oxide, zinc oxide and iron oxide nanoparticles increased thermal stability, impact resistance and adhesion strength, respectively. Also, Muvinkumar et al. [13], studied the effect of nanoparticles and microparticles on the performance of epoxy-based nanocoatings. This study showed that the micro and Nano-particles of silica and zinc have a good bond with epoxy resin, and the adhesion and chemical resistance of particle-reinforced coatings are improved

compared to pure epoxy coating.

The aim of the present research was to introduce an optimal coating with the ability to apply in a simple way by using non-expensive materials and equipment, which at the same time has suitable properties in the desired working condition. The coating in this research is epoxy resin which to increase the working range of this coating in higher temperatures, an additive with thermal retardant properties including polyvinyl chloride (PVC) has been used [14]. To enhance the wear resistance of the coating, nanosilica was added in the coating formulation. After applying the coating, its thermal stability, wear resistance and hardness were evaluated.

2. Experimental

2.1. Materials

Colloidal silica nanoparticles (amorphous structure, particle size 20 nm, 30 %wt silica concentration) were purchased from Iran Nanosylis Isatis Industries company. Epoxy resin (EPIKOTE828), hardener (HY917) and PVC (industrial grade S67) were purchased from Resin Sanat Kian Company, Iran. Silane coupling agent, methyltriethoxysilane (MTES), was purchased from Merck, Company.

2.2. Substrate preparation

In this study, carbon fiber-reinforced polymer-based composite was used as substrate. Specifications of resin and fiber used for manufacturing of substrate samples are presented in Table 1. substrate filed consisted of epoxy resin, which was manually blended by stacking 6 layers of carbon fiber to form a composite sheet to minimize the holes between the resin and the reinforcing fiber fabric, it was kept under vacuum for 15 minutes and then placed in baking conditions; the composite baking cycle is at 80°C for 4 hours and at 120°C for 2 hours. Also, the thickness of the composite sheet reinforced with carbon fibers was 1.50 mm.

2.3. PVC mixing with epoxy resin

First, epoxy resin was slowly mixed with different weight percentages of PVC powder and

Table 1- Materials used for preparation of composite as substrate

Material	specifications
Resin Epon 828	Epon 828 epoxy resin
Curing agent	HY 917
Fiber	3k Carbon fabric, Thickness 0.28 mm, Toray T300

was subjected to mechanical high-speed mixing for 4 hours. Then, the hardener was added to the system and was stirred for 2 hours under magnetic stirring. Finally, the prepared formulation was cured in a vacuum oven for 2 hours at 120°C. After curing, the samples were tested by TGA, and the optimal amount of PVC powder was obtained as a factor in improving the thermal resistance of epoxy resin. Table 2 shows the samples coding based on the amount of added PVC powder to epoxy resin.

2.4. Surface modification of colloidal silica nanoparticles with silane agent

At this stage, the colloidal silica nanoparticles was combined with methyltriethoxysilane (MTES) to bond properly with the silane groups on the silica surface. For this purpose, silane agent and colloidal silica nanoparticles were combined with a ratio of (1.2:1). To control the pH and increase the reaction rate, 0.2 %wt. acetic acid was added to the mixture and was stirred under magnetic stirring for 24 hour at 30 °C to perform the hydrolysis reaction. Then, the sample was cured in an oven at 100 °C and then was subjected to FT-IR test for graft analysis.

2.5. Applying the final coating and curing process

To prepare the final coating system, at first, the of epoxy resin is slowly combined with the optimized weight percentage (20 %wt) of PVC powder and subjected to high speed mechanical mixing for 4 hours. At this stage, different weight percentages of surface modified silica nanoparticles were added to the coating field according to Table

3 and subjected high speed mechanical mixing for 3 hours. Finally, hardener was slowly added to the system and the magnetic mixing was continued for 1 hour at low speed to achieve a suitable coating system. After preparation, the coating material was kept in a vacuum oven for 30 minutes for degassing. The coating on the substrates was done manually and before coating, they were washed and dried with acetone for 10 minutes for cleaning. The coated substrates were dried for 1 hour at ambient temperature and then subjected to final curing for 2 hours at 120 °C. The procedure for the preparation of the coating and its application on the surface are schematically presented in Fig. 1.

2.6. Characterizations

In order to qualitatively study the chemical bonds in the modification of colloidal nanosilica surface by silane agent, FT-IR analysis of samples with and without surface modification was performed using FT-IR instrument model VERTEX 70. The thermal stability of the coatings was evaluated by Q600 Thermo-Gravimetric Analyser. The heating rate of the samples in the air atmosphere is from ambient temperature to 700 °C with a heating rate of 10 min/°C. To test the wear resistance of the samples, the Taber device was used according to D4060 standard method. The abrasive type is used using a CS-10F abrasive wheel under a load of 250 g. A 10 x 10 mm composite substrate with a 7 mm diameter hole in the centre of the sample was used for the test. Hardness testing is used to determine the stiffness of composite samples with and without coating according to ISO15184 standard.

Table 2- Samples coding based on the weight percentages of PVC powder in the coating formulation

Sample	Resin and Hardener (%wt)	PVC (%wt)
E-PV 0	100	0
E-PV 5	95	5
E-PV 10	90	10
E-PV 20	80	20
E-PV 40	60	40

Table 3- Different values of colloidal nanosilica surface modification in the coating system

Sample	E-PV20 (%wt)	Surface modification of nanosilica (%wt)
SM5	95	5
SM7.5	92.50	7.50
SM10	90	10
SM12.5	87.50	12.50
SM15	85	15
SM17.5	82.50	17.50

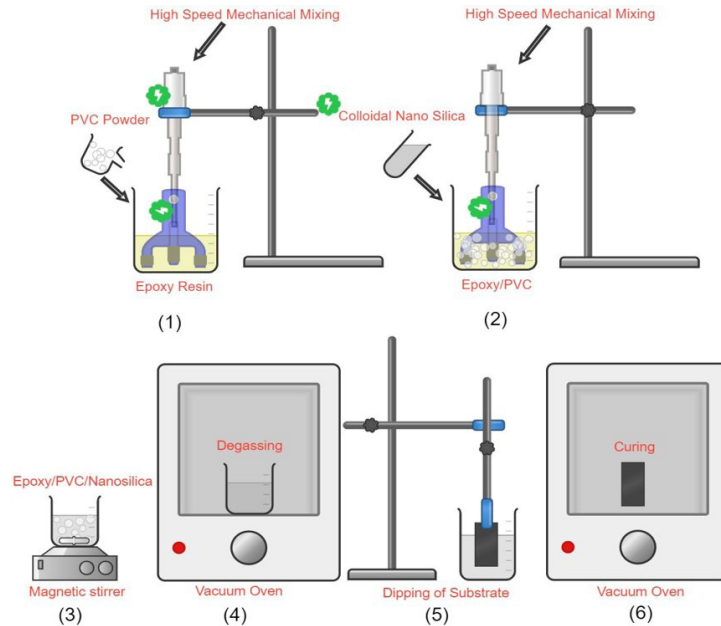


Fig. 1- Schematic representing the coating preparation and its applying on the surface.

Table 4- Thermal degradation temperature of epoxy resin containing different weight percentages of PVC.

sample	10% weight loss (°C)	50% weight loss (°C)	70% weight loss (°C)
EPV0 (A)	275	327	360
EPV5 (B)	252	314	334
EPV10 (C)	266	329	351
EPV20 (D)	331	359	376
EPV40 (E)	287	328	364
PVC	251	283	422

3. Results and discussion

3.1. Effect of adding polyvinyl chloride to epoxy resin

Pure epoxy resin is completely decomposed in the single-stage degradation process in the temperature range between 275 and 400°C. PVC used as a reinforcement in this study has a two-step degradation process. The first stage is observed in the temperature range between 251 and 335 °C, which is associated with the simultaneous creation of double bonds in the polymer chain due to the autocatalytic separation of hydrogen chloride. The second stage involves the decomposition of cross-links in the temperature range of 400 to 460 °C [15].

Figure 2 shows the TGA results for pure epoxy and epoxy/PVC formulation. Table 4 also contains general information on the sample degradation temperature up to 700 °C and the weight loss of the samples in the range of 10 to 70 %wt. As shown in Figure 2 and Table 4, the addition of 5 and 10 %wt of PVC powder to epoxy resin reduces the degradation

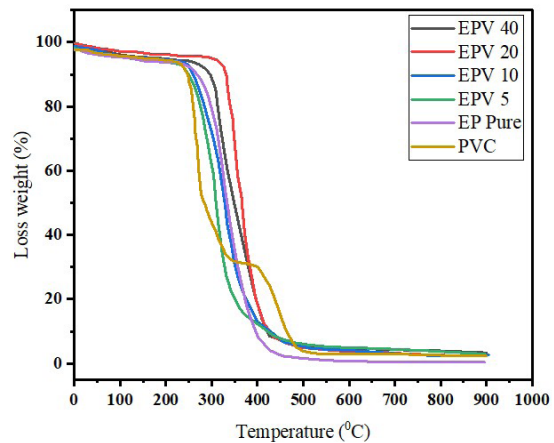


Fig. 2- TGA curves of reinforced epoxy resin with different weight percentages of PVC powder.

temperature of epoxy resin [16, 17], which is associated with a weight loss of 10%. While the addition of 20 and 40 %wt of PVC caused a significant increase in the initial degradation temperature, it was accompanied by a weight loss of 10%. Also,

according to Table 4, in samples containing 20 and 40 %wt of PVC, weight loss of 50% is observed at 359 and 328 °C temperatures, respectively.

The reason for the increase in thermal resistance of epoxy resin in samples containing 20 and 40 %wt of PVC can be attributed to the presence of an additional stage of the decomposition process in PVC powder [16, 17] which is due to mixing with epoxy resin, it has increased the degradation temperature. This effect is due to the relatively high amount of chlorine in PVC, which makes it more resistant to combustion [15-17].

Comparing the resin degradation temperature containing different weight percentages of PVC in Table 4, it is concluded that the neat epoxy sample has an initial degradation temperature of 275 °C; however, the initial degradation temperature of the samples containing 20 and 40% by weight of polyvinyl chloride has the initial degradation temperature of 331 and 287°C, respectively. Therefore, it is obvious that the amount of 20% by weight of polyvinyl chloride powder should be selected as the most optimal amount of additive to increase the thermal resistance of the resin.

3.2. Surface modification of colloidal silica nanoparticles

FT-IR analysis has been used to qualitatively study the chemical bonds in modification of colloidal nanosilica surface with MTES silane agent. Figure 3 shows the FT-IR spectra for the reference sample and the modified sample. According to FT-IR spectrum from the reference sample [18], the Si-CH₃ group in MTES is observed with a strong bond in the range of 1260 cm⁻¹ and also with one or more strong bonds in

the range of 870 to 750 cm⁻¹. The Si-O-C₂H₅ group is observed with weak bonds in the range of 1170 to 1160 cm⁻¹ and also with other weak bonds in the range of 1100, 1075 and 970 to 940 cm⁻¹. Between 1100 and 1130 cm⁻¹, one or more very strong siloxane bonds (Si-O-Si) were observed. As the siloxane chain becomes longer or more branched, the intensity of Si-O-Si absorption becomes more complex, indicating two or more bonds that overlap. In other words, the broadening and complexity of the bond indicates the formation of higher and more branched siloxane chains [19]. The 3470 cm⁻¹ wide absorption band and the 1629 cm⁻¹ absorption band are also related to the O-H bond. By comparing the modified surface sample and the reference sample according to Figure 3, it can be claimed that the results are consistent.

Analysis of the reaction medium by FT-IR shows that the hydrolysis reaction occurred and the corrective reaction was observed about 3 hours after the start of the process. The intensity of the Si-O (H) peak was observed at 900 cm⁻¹. The presence of strong Si-O-Si bonds in the asymmetric stretching state of the 1130 and 1100 cm⁻¹ range and the symmetrical Si-O-Si longitudinal stretch in the 1110 cm⁻¹ range and the Si-O-Si symmetrical stretch in the 870 cm⁻¹ indicates that minor surface modification has occurred. Therefore, it can be concluded that the silane modification was performed by free silane groups within 24 hours.

3.3. wear resistance

Figure 4 shows the wear resistance of the epoxy/carbon substrate without coating and with surface modified coating containing different weight percentages of colloidal silica nanoparticles. It is clear

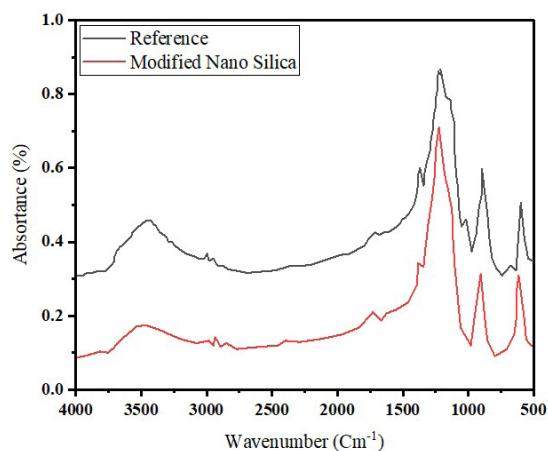


Fig. 3- FT-IR spectrum of reference and colloidal modified colloidal Nano silica

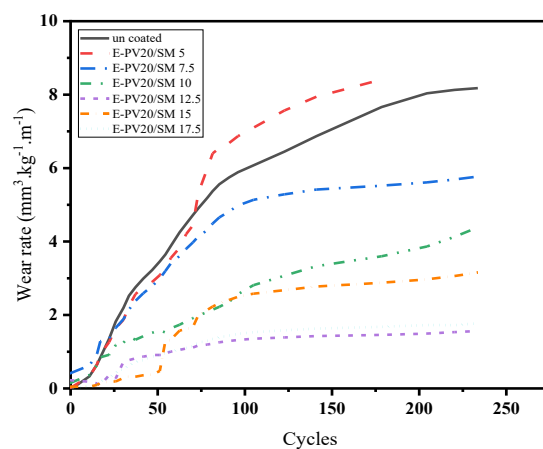


Fig. 4- Epoxy/carbon substrate wear resistance with and without coating.

that the wear rate in different number of wear cycles for epoxy/carbon substrate with a coating containing 12.5 %wt surface modified silica nanoparticles is significantly less compared to substrates with a coating containing 5, 7.5, 10, 15 and 17.5 %wt.

Adding MTES (coupling agent) to colloidal silica nanoparticles (inorganic phase of the coating) and modifying its surface significantly increases the toughness of the coating by forming siloxane chemical bonds between MTES and inorganic particles. Also, the increase in wear resistance due to silane behaviour reduces the surface energy of the inorganic phase, which reduces the possibility of cracking in the coating after curing [20, 21]. Therefore, it can be concluded that the layer of organic-inorganic composite coating consisting of epoxy resin containing colloidal silica nanoparticles with surface modification with MTES, forms a dense siloxane network between the particles and thus shows good wear resistance.

It can also be seen as shown in Figure 4 that the wear rate increases as the percentage of colloidal silica nanoparticles modified to more than 12.5% by weight increases. The reason for this phenomenon can be attributed to the agglomeration and non-uniform dispersion of silica nanoparticles, surface modification in the field of coating with increasing percentage of reinforcing weight [13], which as a defect reduces the desired properties. Therefore, it should be noted that adding a certain amount of surface modified nanosilica to epoxy resin increases the wear resistance and if this value exceeds the critical level, it reduces the wear resistance.

3.4. Hardness

Hardness is a very important factor in the durability

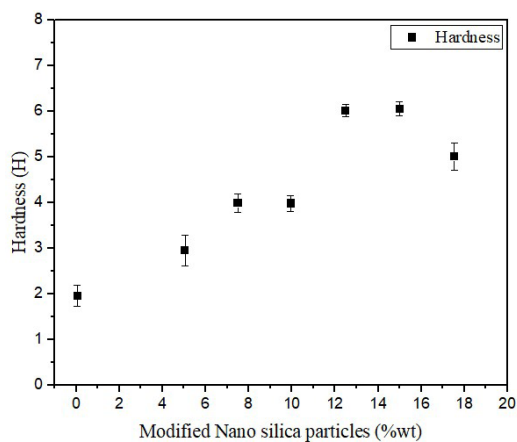


Fig. 5- Hardness of substrates with and without coating.

of coatings [22]. Figure 5 shows the hardness of the substrates with and without coating. As shown in Figure 5, the hardness of the coated epoxy/carbon substrate is increased by adding different weight percentages of the surface modified coating. The hardness of the uncoated substrate is equal to 2H; while the hardness in the coated substrates is 15 and 12.5% by weight of the modified Nano silica is equal to 6H. This significant increase in the hardness of the coated substrates can, firstly, be due to the presence of silica inorganic nanoparticles with hard properties and also a positive effect of surface modification of these nanoparticles with a silane coupling agents [13, 22]. This hardness also indicates the very high adhesion properties of the coating to the composite substrate. With this difficulty, no separation has been observed in the applied coating layer.

Due to the complexity of the wear resistance properties of coatings, the hardness criterion may be used to indicate high wear resistance. Therefore, in this study, by comparing the results of the hardness test of the coated substrates, the results of the wear resistance test can be confirmed, although the data are not always fully consistent.

3.5. Thermal stability

Since the substrates coated with E-PV20/SM12.5 and E-PV20/SM15 had the highest level of wear resistance and hardness, the thermal stability of these substrates was also evaluated in comparison with the substrate without coating. Figure 6 shows the thermal analysis test of the desired substrates. As can be seen, the non-coated epoxy/carbon substrate initially exhibits good thermal stability at temperatures up to 300 °C. Above this temperature, a severe weight loss of approximately 323 °C is

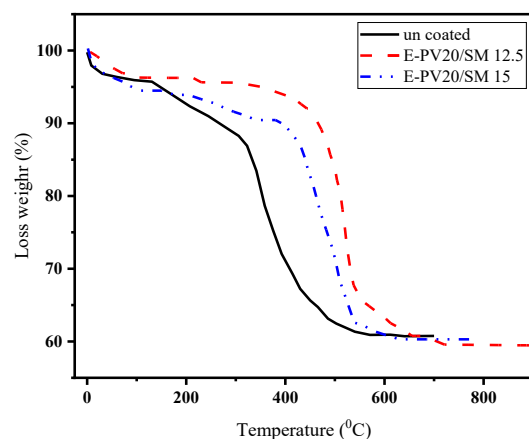


Fig. 6- Epoxy/carbon substrate thermal stability with and without surface modified coating.

observed. At 409 °C, the weight loss was about 30% of the epoxy/carbon composite, at this temperature, almost all the field resin is completely burned and only the carbon fibers remain.

The curve of the epoxy/carbon substrate with a coating containing 15 %wt of modified silica nanoparticles shows a weight loss of 10% at 350°C, which continues until 400 °C. At 503 °C, this curve shows a weight loss of 30%. The curve for the epoxy/carbon substrate containing 12.5 %wt of modified silica nanoparticles shows an initial degradation temperature of about 472°C. Above this temperature, a weight loss of 30% occurs with a starting temperature of 534°C. By comparing all three curves, it is clear that the epoxy/carbon substrate containing 12.5% modified silica nanoparticles has a higher thermal stability than the coated substrate with 15 %wt of the modified silica nanoparticles as well as the non-coated substrate.

The thermal degradation temperature of the coating field has increased with the addition of modified silica nanoparticles, which has also caused the thermal stability of the coated substrate to be higher than that of the non-coated substrate. The significant point is related to the amount of modified silica nanoparticles, when its value was 15 %wt, the initial degradation temperature decreased compared to 12.5 %wt. It can be concluded that the thermal stability also depends on the amount of modified nanoparticles added [23]. It has also been reported that reducing the density of cross-links reduces the degradation temperature of polymer coatings. Therefore, the reason for the decrease in thermal stability of a substrate coated containing 15 %wt of silica nanoparticles can be attributed to this phenomenon. This even reduces the toughness of the failure of the desired coatings. Also, the increase in stability is due to the improvement of the coating barrier property, where the mineral phase can act as a radical to prevent the decomposition of polymer chains [24, 25].

4. Conclusions

Organic/inorganic hybrid coating increase wear resistance and reduce friction coefficient and improve their thermal stability. The presence of nanoparticles on the surface of the coating layer increases the wear hardness and therefore protects the coating layer and the underlying layer of both against cracking damage and soft and hard wears, and also increase the degradation temperature of

the coated substrates. The results of this study are summarized as follows:

1) By adding the optimal amount 20 %wt of PVC powder, the initial degradation temperature of epoxy resin has increased from 275°C to 331°C.

2) The wear rate in the un-coated substrate under load of 250g after 100 wear cycles, from 6 (mm³ .kg⁻¹ .m⁻¹) to 1.5 (mm³ .kg⁻¹ .m⁻¹) in the coated layer with 12.5% wt of modified colloidal nanosilica has decreased, indicating an increase in the wear resistance of the coated substrate compared to the un-coated substrate.

3) The hardness of the un-coated substrate has increased from 2H to 6H in the substrates coated with reinforced of 12.5 and 15% wt of modified silica coatings.

4) The thermal stability of the un-coated substrate was 323 °C; While the thermal stability of the substrate containing the coating reinforced with 12.5 %wt of surface-modified nanosilica was 470°C, which increased by 31.27% compared to the uncoated substrate.

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