Effect of nanoparticles of titanium dioxide in technique textiles

Ana Ma Paniagua Mercado 1,3, Angélica Mauro Nolasco 2, Concepción Mejía García 1, Elvia Díaz Valdés 1 and Joaquín Ibarra Báez 1

1 Escuela Superior de Física y Matemáticas, Instituto Politécnico Nacional, Av. IPN S/N, Col. Lindavista, C.P. 07738, Cd. de México, México
2 Escuela Superior de Ingeniería Textil, Instituto Politécnico Nacional, Av. IPN S/N, Col. Lindavista, C. P. 07738, Cd. de México, México
3 Author to whom any correspondence should be addressed.

E-mail: palianita58@gmail.com

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Abstract

Samples of ceramic fiber textile, with glass insert that supports 640 °C, were characterized. Samples were prepared with 5% and 10% of titanium dioxide nanoparticles, respectively, through starch impregnation method; later they were burned at different temperatures (400 °C, 700 °C, 800 °C and 1100 °C) during 24 h. The weight of each one was obtained, and it was observed that some samples lost more weight than the others. Friction test was conducted to evaluate wear. Macroscopic observations indicated that with 5% TiO2 nanoparticles, the samples were not eroded 100% as the others without nanoparticles, but after being burned above 700 °C an opaque film is formed, and at 1100 °C they became slightly yellowish and diffused corresponding to a change from clinoenstatite to MgSiO3; the samples become brittle from 800 °C. The phases formed and the effect of TiO2 nanoparticles at the surface was determined by XRD, EDS-X analysis. Finally, oxides formed in the fiber at different temperatures were quantified.

1. Introduction

Technical textiles currently have many applications with or without nanoparticles of metal oxides or polymers that are applied from the manufacture of the textile, by impregnation, spray or any other technique where the nanoparticles form a light layer that modifies the thermal, mechanical, optical properties, etc [1]. In this research, the mechanical and thermal properties are the most important, since this refractory textile fiber is an insulator that only supports 640 °C, because it contains a glass insert that melts at that temperature, therefore, it was coated with titanium dioxide nanoparticles. This textile fiber is used as a coating on griddles or plates where radiant heat can cause severe burns to workers who work nearby [2].

The nanoparticles of metal oxides as TiO2 and Ceramics are also used in textile finishing to alter surface properties and impart textile functions with nanosized particles because do not affect the color and brightness when the film is thin and, there are not cumulus of nanoparticles. It was reported, the textiles that are treated with TiO2 nanoparticles, replaced fabrics with active carbon, because the photocatalytic activity of titanium dioxide can attack dangerous and toxic chemicals; those nanoparticles are adhered to textile substrates, converting this textile in a new material [3].

In this research, the adhesion was made by impregnation of nanoparticles of titanium dioxide(nPTiO2) in fiber textile ceramic which is a n-type semiconductor, light sensitive, that absorbs electromagnetic radiation, especially in the UV region; moreover, it is a very stable chemically amphoteric oxide. Due to these characteristics, it is the most commonly used photocatalyst to degrade organic molecules during water purification. It is also used as white pigment, anticorrosive coating, gas sensor, UV absorber and, very especially, it is widely used in the industry of ceramics in whitening and refractories [4], which is where it has many uses as refractory material due to its high melting point (1830 °C) and its thermal properties. Currently for titanium dioxide, it has been found greater application as nPTiO2 nanometrics, especially in fluxes for welding, and...
refractory ceramics fibers, among others. Particularly in this work, they are used as coating in ceramic fiber (insulating) high temperature, as anatase. Besides, when applied as a reinforcing, its reflective properties avoid energy losses and can be applied in insulation of industrial furnaces, and also in industries as the petrochemical, ceramic, the siderurgy and the glassware industry. The thermal properties vary according to each material and this can be partly explained by the movement of electrons and phonons as discrete elastic waves caused by the vibration of the structure. In refractory fibers, efforts are developed because of the thermal expansion of the materials by temperature changes, therefore, it is necessary to be careful in processing and material selection to avoid failures due to thermal stresses [4].

The ceramics fibers as produced are vitreous (glassy) materials which do not contain crystalline silica. If they are exposed at elevated temperatures, this may cause that the fibers become crystalline (devitrify). The first crystalline formation (Mullite) begins to occur at approximately 985 °C. The crystalline phase of silica may begin to form itself at 1100 °C, approximately. When the glass fibers devitrify, they form the mineral crystalline silica. This is trapped in grain boundaries within a matrix that consists predominantly of Mullite. The occurrence and extent of crystalline phase formation is dependent on the duration and exposure temperature, fiber chemistry and/or the presence of fluxing agents or furnace contaminants[5]. All papers related to textiles with TiO$_2$ in the surface, refer to the main properties of TiO$_2$ and its application, describing them as technical textiles of last generation, since before the heating, it could be inserted electronic material and obtain electronic or electrical signals [6]. With the use of photocatalytic characteristics of nanoparticles of TiO$_2$, researchers have imparted new properties to textiles. Self-cleaning, UV protection, antimicrobial qualities, modification of the physical disadvantages of fabrics are some important characteristics that researchers have focused on [7].

The main objective in this work is to obtain a new technique textile, with better mechanical properties, more resistance to the glass insert temperature, better thermal properties of ceramic fibers, a surface more resistant to time-temperature and UV rays.

2. Methods

Experimentation was performed by impregnating nanoparticles of TiO$_2$, in textile samples of 10 cm x 10 cm, with several concentrations of TiO$_2$ in micrometric (0.2 μm–0.3 μm) and nanometrics sizes (21 nm), immersing the ceramic fiber textile, table 1, in a starch aqueous solution at room temperature.

The ratio of starch to water was of 45 g of corn starch and 1240 ml of water. This ratio provides the proper consistency to avoid the precipitation of titanium dioxide, mixing 5% and 10% TiO$_2$ because it is desired to form a protective film in the textile with which they are improved many of their properties as the increase of the thermal resistance of the insert which limits the use of fiber; titanium dioxide will provide many beneficial uses to textile fiber and is insoluble. An aqueous solution of starch is identified as soln. A. The textile is passed through a bath containing only soln. A. and these samples correspond to Group 1. The following samples, Group 2, are prepared by adding 10% TiO$_2$ (with micrometric size) to the soln. A. Samples of Group 3 contain 5% nPTiO$_2$ added to soln. A. Samples of Group 4 contain 10% nPTiO$_2$ in soln. A. All samples are dried outdoors and they are burned at 400 °C, 700 °C, 800 °C and 1100 °C during 24 h. All samples were characterized by texture analysis, color, abrasion, viscosity, x-ray diffraction, macro photographs and EDS-X.

3. Results and discussion

In figures 1 to 4 are shown macrographs of textiles impregnated and burned.

The composition in mass percentage of the ceramic fiber textile contains the elements of the refractory materials Al$_2$O$_3$ and SiO$_2$, as the highest, while the Na, Mg and Ca are accessory elements that have a low melting point that affect the refractory properties of the textile and can be impurities Table 1. The high percentage of Si is due to the insert that is formed by SiO$_2$ in the fiber, which gives the mechanical properties and formation of mullite when reacts with the alumina in the fiber.

The samples of group 1 are identified as M-1- M-2, M-3 and M-4. The sample M-1 was exposed at 400 °C during 24 h without change in coloration; the fabric flexibility to touch, decreases slightly compared to the original sample. The sample M-2 exposed at 700 °C for 24 h presents slight darkening; its flexibility to the touch, also decreases slightly. The sample M-3 exposed at 800 °C for 24 h changes its color to a yellowish blue; its flexibility is more affected compared with the other samples; it is embrittled in both ways warp and weft. The surface of sample M-4 exposed at 1100 °C for 24 h, turns yellowish and molten filaments glass insert (bright filaments) are observed, that in samples exposed to lower temperatures are not presented; the flexibility to the touch, in the direction of the warp and woof, disappears completely, it means that is entirely embrittled, namely, the insert of glass is molten and it must be handled carefully to avoid fractures. This reduced flexibility and increased brittleness with temperature is mainly due to the temperature limit of the glass insert, 640 °C. The glass...
insert improves the mechanical properties; however, at higher temperatures than 640 °C, glass suffers physical and structural transformations and, consequently, the ceramic fiber loses flexibility and ductility, but not burned.

The samples of group 2 are identified as M-5, M-6, M-7 and M-8. The sample M-5 with a base finish and burned at 400 °C does not change its coloration, but a layer is observed in the form of veil with suspended particles, indicating excess of TiO₂. In this sample, the tissue consistency is preserved, although, the threads of wefts and warp exhibited less cohesion, compared to room temperature samples. The sample M-6 exposed at 700 °C for 24 h changes its color to a yellowish hue, due to burning; on its surface a thin layer with suspended particles is observed; this sample presents consistency changes and its flexibility diminishes to the touch, in the direction of the warp and woof, compared with sample M-5. The sample M-7 was exposed at 800 °C for 24 h without change in coloration. Because of the layer of TiO₂ was more refractory to higher temperature, it avoided
changes with the temperature of the layer. This layer contains suspended particles and it is observed in the form of a veil denser than in sample M-6; its flexibility to the touch, is similar to sample M-6. The surface of sample M-8 exposed at 1100 °C for 24 h is entirely yellowish because the organic material is burned, and a layer in the form of veil with a lot of suspended particles is observed; this sample of ceramic fiber loses its initial flexibility,
Table 2. Phases identified by XRD in impregnated and burned textiles.

| Compounds/sample         | M-1 | M-2 | M-3 | M-4 | M-5 | M-6 | M-7 | M-8 | M-9 | M-10 | M-11 | M-12 | M-13 | M-14 | M-15 | M-16 |
|--------------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|------|------|------|------|------|------|------|
| Alumina                  | x   | x   | x   | x   | x   | x   | x   | x   | x   | x    | x    | x    | x    | x    | x    | x    |
| Silica                   | x   | x   | x   | x   | x   | x   | x   | x   | x   | x    | x    | x    | x    | x    | x    | x    |
| Clinoenstatite           | x   | x   | x   | x   | x   | x   | x   | x   | x   | x    | x    | x    | x    | x    | x    | x    |
| Titanium dioxide         |     |     |     |     |     |     |     | x   | x   | x    | x    | x    | x    | x    | x    | x    |
| Magnesium silicate       | x   |     |     |     |     | x   |     |     |     |     |     |     |     |     |     | x    |
| Cellulose                | x   |     |     |     |     | x   |     |     |     |     |     |     |     |     |     | x    |
| Mullite                  | x   |     |     |     |     | x   |     |     |     |     |     |     |     |     |     | x    |
due to transformation of glass to another phase. The sample M-7 was exposed at 800 °C for 24 h without change in coloration.

The samples of group 3 are identified as M-9, M-10, M-11 and M-12. The sample M-9 was burned at 400 °C for 24 h, it was observed that the part where the samples were cut, it became dark, indicating a slight starch burn. In the surface a layer is observed in the form of veil with suspended particles, less dense than for group 2; the texture and flexibility in direction of the warp and woof are not affected because the glass insert remains unchanged. The sample M-10 was exposed at 700 °C for 24 h without change in coloration due to the introduction of nanometrics particles between fibers of the material, a thin veil of nanoparticles of TiO₂ was formed on the surface with an accumulation of suspended particles, indicating an excess of nanoparticles; as in sample M-9, the texture and flexibility in direction of the warp and woof are not affected because the glass insert remains unchanged, namely, a protective layer more refractory was formed with TiO₂ nanoparticles, causing the ceramic fiber surface more resistant to the temperature and thus protecting the glass insert.

Sample M-11 exposed at 800 °C for 24 h presents slight change in coloration; it is observed also a veil less dense compared with samples of group 2; its flexibility slightly decreases in the direction of the warp and woof, because the glass insert that gives it the mechanical properties, at this temperature maybe begins its fusion.

Sample M-12 exposed at 1100 °C for 24 h changes its color to a yellowish hue; at the surface is observed less agglomeration of nanoparticles than in the veil formed in the sample M-11. Because of the glass insert has already begin melted it changes its texture, it is more brittle and must be handled carefully to avoid causing fractures. The samples of group 4 are identified as M-13, M-14, M-15 and M-16. Sample M-13 was exposed at 400 °C for 24 h, it became dark on the banks where the sample was cut, and loss of fibers were generated due to starch burnt. In this sample also is obtained a veil on the surface and nanoparticles suspended due to excess of them; it shows good flexibility in direction of the warp and woof. Sample M-14 exposed at 700 °C for 24 h changes to a darker and yellowish color; more particles, by accumulation, are observed on the surface of the veil, compared to the above samples; its flexibility in direction of the warp and woof is maintained, due to refractoriness of titanium oxide. The sample M-15 exposed at 1100 °C follows the change pattern of hue when temperature increases; the coverage area is greater because a dense veil with nanoparticles suspended is formed, although, because of the excess of nanoparticles of TiO₂ detachment occurs; the sample can be manipulated without difficulty, indicating that, the glass insert remains protected by nanoparticles and it has not melted. The sample M-16 exposed at 1100 °C presents a veil on its surface with agglomerations where the pigmentation is

| T(°C)/%W | G-1   | G-2   | G-3   | G-4   |
|----------|-------|-------|-------|-------|
| 400      | 16.17 | 15.90 | 15.58 | 15.21 |
| 700      | 14.39 | 29.44 | 27.39 | 28.00 |
| 800      | 26.47 | 15.86 | 16.32 | 19.41 |
| 1100     | 17.28 | 20.61 | 19.10 | 22.14 |

Figure 5. Weight loss or gain % versus temperature of samples of all groups after burning.
darker by organic matter, with a burned appearance; the mechanical strength of the sample is decreased, so that, it should be handled carefully to avoid fractures, since, the yarn loses consistency because at this temperature the glass insert is melted.

3.1. X-ray diffraction

The samples at room temperature and 400 °C showed the reflections of alpha alumina ($\alpha$-Al$_2$O$_3$), silica (SiO$_2$), cellulose ((C$_6$H$_{10}$O$_5$)$_x$) and clinoenstatite (MgSiO$_3$). The principal compounds are Alumina, silica, and clinoenstatite at 400 °C, 700 °C, and 800 °C, but at 1100 °C, the clinoenstatite is transformed into magnesium silicate which is the same composition of the clinoenstatite.

Samples impregnated with TiO$_2$ nanoparticles, as is expected, presented the reflections of TiO$_2$. The samples burned at 700 °C and 800 °C showed the peaks of the oxides but not the peak of the cellulose, because it is melted at 460 °C. In the samples burned at 1100 °C are observed alpha alumina and silica; the clinoenstatite is transformed to magnesium silicate and traces of mullite phase appear. With the formation of mullite, the fiber becomes more refractory, that is to say, it supports more temperature, is more resistant to thermal shock and improves its mechanical properties, as observed, in textiles coated with nanoparticles. The only material that disappears is the starch, the other phases that form the ceramic fiber kept the same composition, including the glass of the insert does not disappear only change its crystallization. The nanoparticles of TiO$_2$ continue adhered to the samples over the filaments of the fibers, as they were deposited.

| Table 4. Weight loss % in Abrasion Test. |
| T(°C)/%W | G-1 | G-2 | G-3 | G-4 |
|----------|-----|-----|-----|-----|
| 400      | 100.00 | 46.34 | 64.85 | 35.06 |
| 700      | 49.28  | 54.07 | 85.34 | 100.00 |
| 800      | 100.00 | 59.94 | 20.42 | 62.11 |
| 1100     | 100.00 | 85.61 | 41.14 | 29.66 |

| Table 5. Chemical Composition by EDS-X of ceramic textiles impregnated with TiO$_2$. |
| Sample/%| C | O | Na | Ti | Mg | Al | Si | Ca |
|---------|---|---|----|----|----|----|----|----|
| M -1    | 3.9| 47.1| 0.1| —  | 0.7| 13.8| 32.3| 2.0 |
| M-2     | 23.1| 39.6| 0.2| —  | 0.6| 16.2| 18.4| 1.9 |
| M-3     | 12.9| 41.9| 0.2| 1.0| 0.7| 14.3| 26.6| 2.5 |
| M-4     | 28.6| 36.7| 0.3| 1.9| 0.7| 13.4| 15.6| 3.0 |
| M-5     | 34.8| 35.2| 0.2| 0.9| 0.5| 13.5| 13.8| 1.2 |
| M-6     | 15.0| 41.2| 0.2| 1.23| 0.67| 19.1| 20.5| 2.3 |

Figure 6. Abrasion test % versus temperature of all samples of by groups after burning.
Table 2 resumes the phases identified by XRD.

3.2. Mechanical properties
The mechanical properties were measured in samples with and without impregnation and with and without burned by applying abrasion tests. In abrasion tests, samples burned at 400 °C, 700 °C, 800 °C and 1100 °C with and without impregnation, subject to wear 24 h, were evaluated. In samples 5 × 5 cm were measured which percentage of the sample resisted the abrasion. The samples with and without abrasion test, [8] were weighed, as well as, the residue obtained. The last one is affected by the test and indicates the deterioration of the mechanical properties of the textile fiber.

3.3. Abrasion test after burning
Table 3 presents the weight loss % after burning for samples of groups 1 to 4. Figure 5 shows the graph of the weight loss % versus temperature for all groups after burning.

The sample that lost more weight was the M6 of the G-2 at 700 °C with 29.44%, which could be due to the fact that the particle size of TiO2 for all those samples of group 2 was in microns, much bigger than the size of the nanoparticles.

The lowest percentage lost was found in sample M-2 of G-1 calcined at 700 °C and without TiO2.

3.4. Weight loss in abrasion test
Table 4 presents the weight loss % of G-1 to G-4 after abrasion test and in figure 6 is shown its graph.
Figure 9. Sample M-2, 4500 X.

Figure 10. EDS-X Sample M-2.

Figure 11. Sample M-3, 5000 X.
Figure 12. EDS-X Sample M-3.

Figure 13. Sample M-4, 14000 X.

Figure 14. EDS-X Sample M-4.
The textile material that best resisted the abrasion test and remained in better condition was the M-11 G-3 with only 20.42% of weight loss; this is evidence that the insert was undamaged. This sample was impregnated with 5% of nanoparticles and calcined at 800 °C.

3.5. Wear test
Of the samples exposed at 400 °C, the experiment with the least wear showed 50.71% wear; this was the sample M-5 impregnated with 10% of TiO$_2$ without grinding. The samples exposed at 700 °C, 800 °C and 1100 °C, wear out completely. The samples exposed at 700 °C had an average wear of 46% except for the M-14 with a wear of 18.56%. At 640 °C, the glass insert is broken, therefore, the strength properties of the fabric are seriously affected; however, they benefit from the application of the finish.

The samples exposed at 800 °C, in particular, the M-3 and M-15, had an average wear of 47%, while the M-7 had the best performance in abrasion tests at this temperature. The sample impregnated with 10% of titanium dioxide without grinding, presented a 14.65% of wear. Analysis of the samples burned at 1100 °C, indicate that the sample M-4 had 64.93% of wear; the sample M-8 of 100%, while the sample M-12 showed 37.88% and the sample M-16 presented 70.33% of wear. In order that, the textile fiber can keep its flexibility, the service temperature of the textile fiber—ceramic, with glass insert, must be 649 °C. At 1100 °C the glass insert is broken.

During this abrasion test, sample with the best performance was the M-12 with 5% Titanium dioxide nanoparticles, compared to the sample without any finishing.

3.6. Chemical analysis of textiles
Table 5 shows the chemical composition for samples M-1 to M-6 by EDS-X with and without impregnation at different quantities and size of nanoparticles.

M-1 corresponds to normal textile, M-2 to calcined textile, M-3 to textile with 10% TiO$_2$, M-4 to textile with 5% TiO$_2$ nanoparticles, M-5 to textile with 10% TiO$_2$ nanoparticles and, M-6 to textile with 10% TiO$_2$ nanoparticles.

Sample M-1 is the original sample without any treatment, sample M-2 is without treatment but burned at each of the temperatures, M-3 is a sample with 10% TiO$_2$ of micrometric size, that is the commercial size of Titanium dioxide.

In this sample, the 1% of Ti with lower amount of carbon than the sample M-2 is already observed, which indicates that the burning of the textile was smaller.

The analysis of the sample M-4 coated with 5% titanium dioxide nanoparticles gives a higher amount of TiO$_2$ with a larger amount of carbon, that indicates a poor protection.

M-5 has a content of TiO$_2$ equal to M-3 and the highest amount of Carbon and a lower content of elements of low melting point.

M-6 sample was coated with 10% of TiO$_2$ nanoparticles. In the microanalysis the largest amount of Titanium is detected, the carbon decreases indicating a less burned fiber, the Al increases and a slight increase in silicon with respect to the previous two is observed; it is also formed mullite phase.
Chemical Composition, by EDS-X (Table 3) of ceramic textiles with and without TiO2 nanoparticles and in that impregnated fibers textiles with TiO2 nanoparticles with 10%. Sample M-6 was the most impregnated with TiO2 nanoparticles.

3.7. Micrographs and EDS-X analysis of selected samples

Different ceramic fiber textile was selected (Figures 7–18) to which semi quantitative chemical analysis was applied by EDS-X [10], to determine if the TiO2 has normal size (2 μm–3 μm) or nanometrics size (21 nm) and how it reacts with the ceramic textile [11] as well as to determine the composition.

The selected samples were: textile standard burned at 400 °C, M-1 figure 7; calcined textile without TiO2, M-2 figure 9; textile with 10% TiO2 without grinding, figure 11; textile with 5% nanoparticles of TiO2 at 700 °C, M-3, figure 11 textile with 5% nanoparticles of TiO2 at 800 °C, M-4 figure 13; and textile with 10% nanoparticles of TiO2 at 1100 °C, M-5 figure 15. In all samples, the variations were: temperature, size and amount of titanium dioxide, the amount impregnated on the surface to perform the SEM analysis [12], and the amount formed by burning temperature. It was observed that at higher temperatures more carbon is formed (1100 °C).

The textile with more titanium dioxide was the 6, since, 10% of TiO2 nanoparticles were deposited; this means that because of size, the nanoparticles penetrated more in the textile fiber [13], although, the 10% is considered in excess.

In figure 17, the micrograph was 50000X. Until this magnification the particles are observed in nm, but the image loses resolution.
With the impregnation of particles and nanoparticles of Titanium Dioxide, the textile resultant was more resistant to temperature, with better properties for some cases, [14] this mean higher protection to ultraviolet-rays; when the textile is used as cover to ray-sun, making with this a different textile to initial, [15, 16, 17].

4. Conclusions

1. The best thermal properties were obtained all for samples impregnated by nanoparticles of Titanium dioxide, and the majority and acquired protection UV.

2. With the cover of nanoparticles in the surface textile the glass insert until 800 °C, where the material of insert does not break only is phase transformed.

3. The highest quantity of nanoparticles of Titanium dioxide impregnated in the surface of the textile was found in the sample with 5% of nanoparticles and burned at 700 °C.

4. The best mechanical properties was observed for impregnated textile with 10% of nanoparticles and nothing burned at 700 °C. This sample was better for the abrasion test.

5. The temperature resistance too was increase with nanoparticles film and to anything temperature was detected carbon formation in the fibers.

ORCID iDs

Ana Ma Paniagua Mercado @ https://orcid.org/0000-0002-7631-4589

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