

# SOL-GEL SYNTHESIS OF TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO NANOCOMPOSITE

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*The sol-gel process has received a great deal of attention in the past decade due to advantages such as low temperature processing and high homogeneity of final products. The preparation of TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite by the sol-gel method is efficient at producing thin, transparent multi-component oxide layers. In this study, the preparation of TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite and its characterization were investigated. TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO nanocomposite was prepared from tetraethoxysilane (TEOS), titanium n-butoxide (TBO) and GLYMO (3-glycidoxypropyl)-trimethoxysilane) catalyzed with acid. Scanning electron microscopy (SEM) was employed to characterize the surface properties of composite films. The chemical structure of the composite was evaluated by means of Fourier transform infrared spectroscopy (FTIR) and Raman spectroscopy. Particle size was determined by a particle sizer. In summary, transparent and uniform nano colloidal TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite solutions were successfully synthesized through the sol-gel method. The turbidity values of the TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composites were in the range of 8.4±0.4–12.7±0.6 ntu, and the pH values were in the range of 4.8 to 5.2. The particle sizes of the obtained composites were in the range of 5.9±1.3–22.1±1.2 nm. It was determined that the viscosity of the TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite solutions was approximately equal to 3-7 mPas. The thin transparent coatings obtained from these solutions were evenly distributed.*

**Keywords:** TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO nanocomposite, Sol-gel, Leather, Finishing, Coating

## 1. Introduction

The sol gel process has received a great deal of attention in materials research in the past decade due to its unique advantages, such as low temperature processing, high homogeneity of final products and its capability to generate materials with controlled surface properties and pore structures [1]. The sol-gel process is one of the technologies with the best potential for the preparation of TiO<sub>2</sub> (titanium dioxide) photocatalyst. It can be classified as a wet-chemical technique as it uses chemical solutions (sol) as a precursor for an integrated network (gel) of either discrete particles or network polymer in order to fabricate materials (typically metal oxide) [2].

Thin transparent layers containing TiO<sub>2</sub> have been intensively studied due to their interesting application potential in the photocatalytic purification of water and air [3], antimicrobial and self-cleaning functions [4-6]. The physical and chemical property of TiO<sub>2</sub> crystallite size can be controlled by adding a second semiconductor to the TiO<sub>2</sub> matrix. Silicon dioxide (SiO<sub>2</sub>) has been incorporated in the TiO<sub>2</sub> matrix to enhance the photocatalytic process [7]. SiO<sub>2</sub> has high thermal stability and excellent mechanical strength, and helps to create new catalytic active sites due to interaction between TiO<sub>2</sub> and SiO<sub>2</sub>. In addition, SiO<sub>2</sub> acts as the carrier of TiO<sub>2</sub> and helps to

provide a large surface area as well as a suitable porous structure [8].

The use of organic components for the improvement of the properties of TiO<sub>2</sub> layers has begun relatively recently. GLYMO is an organically modified alkoxide, whose organic group contains an epoxide ring that can be cross-linked to form a poly(ethylene oxide) chain and acts, therefore, as a network former. The use of GLYMO could greatly change the structural properties of sol-gel derived xerogels and allow materials with higher density at low temperatures to be obtained [9].

In our previous studies, colloidal TiO<sub>2</sub>-SiO<sub>2</sub> composite solution was successfully synthesized using the sol-gel method [10]. The aim of this study was to prepare TiO<sub>2</sub>-SiO<sub>2</sub> composite with the addition of "3-glycidoxypropyltrimethoxysilane (GLYMO, [(OCH<sub>2</sub>CH)<sub>2</sub>CH<sub>2</sub>OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>]-Si(OCH<sub>3</sub>)<sub>3</sub>) with different molar ratios at ambient temperature through the sol-gel method in order to apply it in the finishing process as a top coat on a leather substrate. Therefore, the turbidity, viscosity and pH of the nanocomposite were measured in order to reveal its characteristics. The surface properties and chemical structure of composite films were characterized using scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) spectroscopy. Particle size was measured using a particle sizer.

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Table 1

Composition by weight of the precursors used						
Sample	TBO (mol)	TEOS (mol)	H <sub>2</sub> O/TEOS+TBO (mol ratio)	Acid/TEOS+TBO (mol ratio)	Ethanol/TEOS+TBO (mol ratio)	TBO/TEOS/GLYMO (mol % ratio)
1	0.1	0.1	2	0.05	6	50/50/0
2	0.1	0.1	2	0.05	6	40/40/20
3	0.1	0.1	2	0.05	6	60/20/20

Table 2

Main characteristics of TiO <sub>2</sub> -SiO <sub>2</sub> -GLYMO composite solutions				
TBO/TEOS/GLYMO (mol ratio %)	Turbidity, ntu	pH	Viscosity, mPa s	Particle size, nm
50/50/-	12.7±0.6	5.2	3.3±0.1	5.9±1.3
40/40/20	9.9±0.8	4.8	6.9±0.4	22.1±1.2
60/20/20	8.4±0.4	5.1	5.7±1.1	10.3±1.3

## 2. Materials and method

### 2.1. Materials

Tetraethyl orthosilicate (TEOS) and (3-glycidoxypropyl)-trimethoxysilane (GLYMO) were all analytical grade and purchased from Sigma-Aldrich (USA). Titanium n-butoxide (TBO) was provided by Merck (Germany). All of these materials were used as received. Distilled water was used for the hydrolysis of alkoxides, and acetic acid (100%) was used as a catalyst. Analytical grade ethyl alcohol was the solvent for all the sols. Acetylacetone was used as a chelating agent.

### 2.2 Preparation of TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite

The TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite solution was produced in the following way [11]: half amounts of the required ethanol, acetic acid and distilled water were mixed in a round-bottomed flask with stirring. The required amount of TEOS was added dropwise to this solution with 30 minutes stirring to partially hydrolyze the TEOS. Then, TBO in acetylacetone was added dropwise to the partially hydrolyzed TEOS solution. A mixture of the other half of the ethanol, acetic acid and distilled water was added to complete the hydrolysis process of the SiO<sub>2</sub> and TiO<sub>2</sub>. GLYMO was added to the TiO<sub>2</sub>-SiO<sub>2</sub> dispersion with the molar ratio given in Table 1. TiO<sub>2</sub>-SiO<sub>2</sub> solution without GLYMO was also prepared to compare the properties. Ultrasonic treatment was applied for 15 min under 15 kW power. The final transparent sol was then aged for three days to obtain dispersion. The molar ratio of TEOS/TBO and (TBO+TEOS)/H<sub>2</sub>O/acetic acid is summarized in Table 1. To investigate the properties of TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO nanocomposite films, 0.3 ml of each composite was poured on to a glass slide (3x7 cm) which was left on a horizontal surface at room temperature until it was dry and a film was formed.

### 2.3 Characterization of TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite films

In order to determine the solution characteristics which affected nanoparticle forma-

tion, the turbidity, pH values and rheological properties of the prepared solutions were measured and the size of particles was determined. The turbidity properties of the solutions were measured for use as standard solutions for the coating process using a Delta OHM Turbidimeter (Italy) according to the ISO 7027 nephelometric method. The measurements were taken in the range of 0–1000 nephelometric turbidity units. After the preparation of the transparent solutions, the pH values of the solutions were measured to determine their acidic and basic characteristics with a standard pH meter (WTW Inolab, Germany). Additionally, the rheological behavior of the solutions, including the viscosity, was obtained with a Rheosys Merlin VR digital rheometer (USA). Particle sizes were determined with a Malvern Mastersizer 2000 (UK). Scanning electron microscopy (Quanta FEG 250, USA), Fourier transform infrared (Perkin Elmer, USA) and Raman spectroscopy (Thermo DXR, USA) were employed to characterize the surface properties and chemical structure of the nanocomposite films, respectively. FTIR and Raman spectra were obtained in the range of 4000 to 400 cm<sup>-1</sup> at a resolution of 2 cm<sup>-1</sup>. Atomic Force Microscopy (XE-Nanomagnetics Instruments, UK) were employed to characterize the surface property of nanocomposite films.

## 3. Results and Discussion

Table 2 presents the turbidity, pH and viscosity values of the colloidal solutions which were prepared. Turbidimetric measurements were made to check for the complete dissolution of the powder-based precursors in the solutions. The turbidity values were in the range of 8.4±0.4–12.7±0.6 nephelometric turbidity units, indicating that the chemical precursors had completely dissolved in the solutions. It is important to note that the pH value of sols is a factor which influences the formation of the polymeric three-dimensional structure of the gel during the gelation process and it should be taken into consideration

when preparing solutions. The pH values of composite solutions have a mildly acidic pH value of 4.8 to 5.2 (Table 2). It was determined that the viscosity of the nano-sized TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO colloidal solutions with different concentrations were in the range of 3.3-6.9 mPa s (Table 2). In our case, nano TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO films were obtained with low-viscosity diluted solutions. A small decrease in the viscosity of solutions depending on the test time probably signals fragmentation of the network as strong association complexes are formed. The particle sizes were in the range of 5.9±1.3–22.1±1.2 nm (Table 2).

It is claimed that one of the parameters governing the morphology of the product is the pH of the forming disperse system. Thus, a change in the pH of the dispersion medium in the final stage makes it possible to reduce the particle size [12]. The pH values of 4.8-5.2 of the solutions obtained in our study had a mild acidic character, which are in agreement with the study where TiO<sub>2</sub>-SiO<sub>2</sub> composite was obtained by adding GLYMO to a nanoparticle dispersion of TiO<sub>2</sub> and it was reported that the pH of the solutions varied between 5.4 and 5.9 depending on the ratio of GLYMO to TiO<sub>2</sub> [13]. These values are within suitable limits both for the formation of Ti-O-Si bonds in the TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite and for the finishing process to be applied to the leather.

It was also found that all three solutions in our study were transparent. If the solid materials in the solution do not dissolve, a transparent solution does not result, and the morphology of the film obtained is not regular. Some researchers reported that small amounts of Ti precursor and low operating temperatures were suitable for obtaining denser and more transparent films [14]. Other authors have concluded that the transparency of the composite arises from the addition of SiO<sub>2</sub> because of the small size of the particles and their high distribution [15]. It was concluded in our study that separate preparation of precursor solutions and their drop by drop addition to form a mixture as well as choosing the sol-gel method for composite synthesis were resulted in obtaining transparent solutions. This issue will give important indications that the composite solutions will not affect the natural appearance of the leather at the stage when it is applied, and about the film which will be formed on the leather in the finishing process.

When the effect of water content in the sol on the characteristics of TiO<sub>2</sub>-SiO<sub>2</sub> composite films was examined, it was revealed that when the water content increased, the sol viscosity and the thickness of the film also increased [16]. The viscosity values determined in the study are within the optimum values for the leather coating process, or in other words, the viscosity values of the composite are appropriate for application to leather by the spraying method. In addition, the sizes of TiO<sub>2</sub> and SiO<sub>2</sub> particles in the composite solutions

are suitable for easy penetration of the pores of the leather. This suggests that the nanocomposite solutions obtained will confer an advantage in the finishing process and will greatly affect various fastness characteristics of the leather including water vapour permeability, since they will provide more free volume (the pores or gaps between nanoparticles and their interface with substrate) for the composite film [17, 18].

To investigate the chemical structure of nanocomposites, the FTIR spectra of TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composites were measured (Fig. 1).

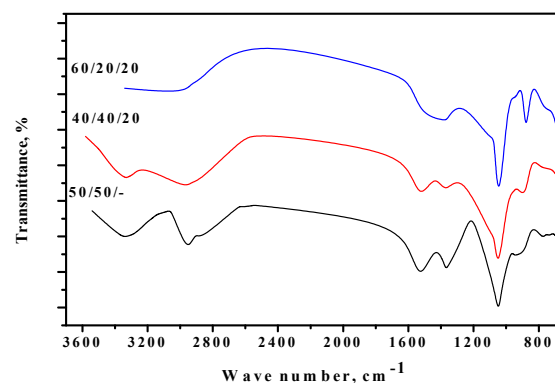


Fig. 1 - FTIR spectra of TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO nanocomposites.

The characteristic peaks of Si-O-Si were present at wavenumbers of 1100 cm<sup>-1</sup> and 805 cm<sup>-1</sup>. Si-O-R and Si-O peaks were observed at 1048 cm<sup>-1</sup> and 1078 cm<sup>-1</sup> respectively. Moreover, a new characteristic peak at a wavenumber of 932 cm<sup>-1</sup> occurs in this case, indicating the formation of new Ti-O-Si bonds. These findings were in agreement with the studies where the peaks at 1000-1250cm<sup>-1</sup> belonged to asymmetric stretching vibrations of the Si-O-Si band, and the peaks between 775 and 440 cm<sup>-1</sup> belonged to symmetric stretching vibration of the Si-O-Si band, while the peaks at a wave number of 935 cm<sup>-1</sup> belonged to the Si-O-Ti and Si-OH bands [19, 20]. In Fig. 1, the peaks at wavenumbers of 1530 cm<sup>-1</sup>, 1432 cm<sup>-1</sup> and 1365 cm<sup>-1</sup> correspond to the characteristic peaks of -CH<sub>2</sub>, while the peaks at a wavenumber of 3100 cm<sup>-1</sup> and 3600 cm<sup>-1</sup> are the characteristic peaks of O-H. For TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite, the peaks at wavenumbers of 2875 cm<sup>-1</sup>, 1527 cm<sup>-1</sup> and 1582 cm<sup>-1</sup> are attributed to the characteristic peak of -CH<sub>2</sub> which was in agreement with some previous studies [21, 22]. Some authors revealed that composite modified with GLYMO formed new characteristic peaks at wavenumbers of 1200 and 1093 cm<sup>-1</sup> belonging to the Si-O-Me group, but when the GLYMO was completely hydrolysed, these peaks did not form. They also reported that the peak forming at a wavenumber of 1050 cm<sup>-1</sup> belonged to the Si-O-Si bond, and this bond indicated the formation of a Si-matrix [13].

Raman spectroscopy was applied to gain

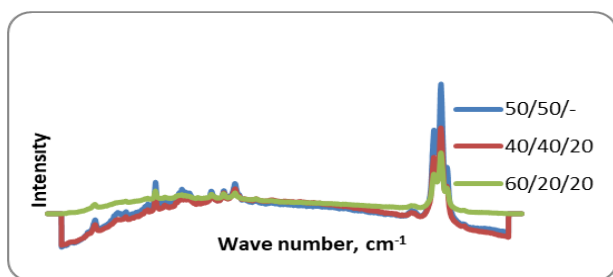


Fig. 2 - Raman spectra of  $\text{TiO}_2\text{-SiO}_2\text{-GLYMO}$  nanocomposites.

better understanding of the structure of the prepared  $\text{TiO}_2\text{-SiO}_2\text{-GLYMO}$  nanocomposite. The Raman spectra of the nanocomposite are given in Figure 2. The sharp peak at  $146\text{ cm}^{-1}$  indicated the presence of  $\text{TiO}_2$  in the anatase phase. Although the rutile phase also exhibits a peak around this area it has very weak signal, therefore this peak showed the presence of the anatase phase [23]. The  $\text{SiO}_2$  peaks are also quite weak and hard to distinguish in the spectra however the broad peak centered at  $300\text{ cm}^{-1}$  in the spectra indicated the presence of a  $\text{SiO}_2$  shell [24].

The SEM micrograph of the surface of the  $\text{TiO}_2\text{-SiO}_2\text{-GLYMO}$  composite film sample (Figure 3a, b, c) reveals that a smooth thin film was obtained on the substrate. In fact, the coating was evenly and homogeneously distributed over the surface. Thus, it was observed that the advantages of the sol-gel method such as good homogeneity, ease of composition control and low processing temperature had been of benefit, and had given thin and evenly distributed films on the substrate. It was clear that the precursors used had dissolved and that the morphology of the films obtained was regular (Figure 3a, b, c).

The  $\text{TiO}_2\text{-SiO}_2\text{-GLYMO}$  composite films were assessed with an atomic force microscope (AFM). AFM analysis showed a composite film thickness of  $7.42\text{-}7.43\text{ }\mu\text{m}$  for  $\text{TiO}_2\text{-SiO}_2$  (50/50/),  $5.20\text{-}5.23\text{ }\mu\text{m}$  for  $\text{TiO}_2/\text{SiO}_2/\text{GLYMO}$  (40/40/20), and  $5.98\text{-}7.89\text{ }\mu\text{m}$  for  $\text{TiO}_2/\text{SiO}_2/\text{GLYMO}$  (60/20/20). The micron thickness of the films arose due to the immersion method by which they were produced. The particles in the  $\text{TiO}_2\text{-SiO}_2$  film were almost all evenly distributed, and were in the form of small particles (Figure 4a), while most of the particles in the  $\text{TiO}_2\text{-SiO}_2\text{-GLYMO}$  film were in large clusters (Figure 4b, c).

The optical properties of  $\text{TiO}_2\text{-SiO}_2\text{-GLYMO}$  composite films were investigated and it was observed in SEM images that films dried at  $100^\circ\text{C}$  were firm, without pores, of  $1.5\text{ }\mu\text{m}$  thickness and amorphous in structure [25]. In AFM imaging, the structure of films dried at  $80^\circ\text{C}$  was reported to be rough and without pores, and this was probably because of the organic solvent remaining in the film. In this way, it was determined that the composite material reached its final density at a

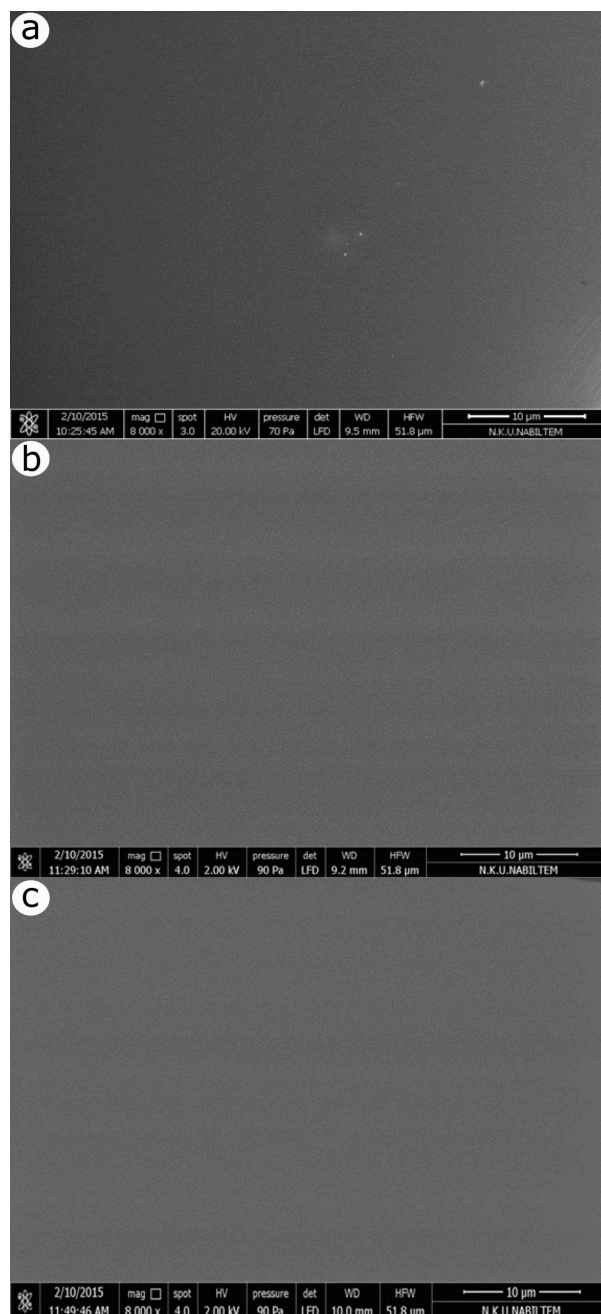


Fig. 3 - SEM micrographs of  $\text{TiO}_2\text{-SiO}_2\text{-GLYMO}$  nanocomposite a) 50/50/0, b) 40/40/20, c) 60/20/20.

relatively low temperature, and this probably arose from the organic components filling the pores between the inorganic oxide chains [26]. In TEM images of  $\text{TiO}_2$  doped  $\text{SiO}_2$  composite films on a glass substrate, the particles formed showed a homogeneous distribution and were spherical in shape, with an average particle size of  $5\text{-}15\text{ nm}$  [27]. Palmisano et al. determined in SEM images that thin  $\text{TiO}_2/\text{ormosil}$  films obtained were  $2.0\text{-}3.2\text{ }\mu\text{m}$  in thickness and homogeneous [28].

In the present study it was determined that most of the particles in the films showed even distribution and most had a spiral appearance. The small size of the particles and their even distribution are the reason why the film covered



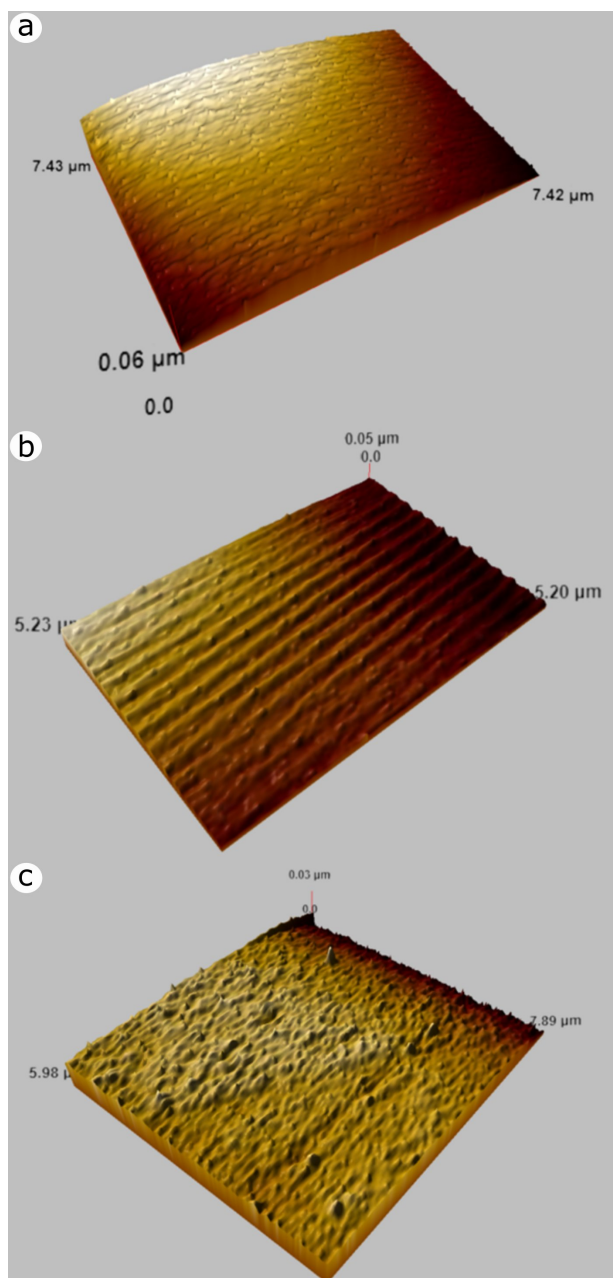


Fig. 4 - AFM micrographs of TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO nanocomposites a) 50/50/0, b) 40/40/20, c) 60/20/20

the substrate in a homogeneous way. Within this framework, the studies were focused on the investigation of the performance of colloidal TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO nanocomposite as finishing coating on leather material. When the TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO nanocomposite was applied on leather with lacquer the performance characteristics of leather such as finish adhesion, dry and wet rubbing fastness, colour fastness to water and perspiration, to light and UV light were improved [29-30].

#### 4. Conclusions

In summary, transparent and uniform nano colloidal TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite solutions were successfully synthesized using the sol-gel method. The turbidity values of the composites

were in the range of 8.4±0.4–12.7±0.6 ntu, and the pH values of the TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO sols were mildly acidic with a pH value of 4.8 to 5.2. The particle sizes were in the range of 5.9±1.3–22.1±1.2 nm. It was determined that the viscosity of the TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO composite solutions was approximately equal to 3-7 mPas. The thin transparent coatings obtained from these solutions were evenly distributed. From the results obtained, it was revealed that the TiO<sub>2</sub>-SiO<sub>2</sub>-GLYMO nanocomposite has favourable properties for use as a leather coating additive.

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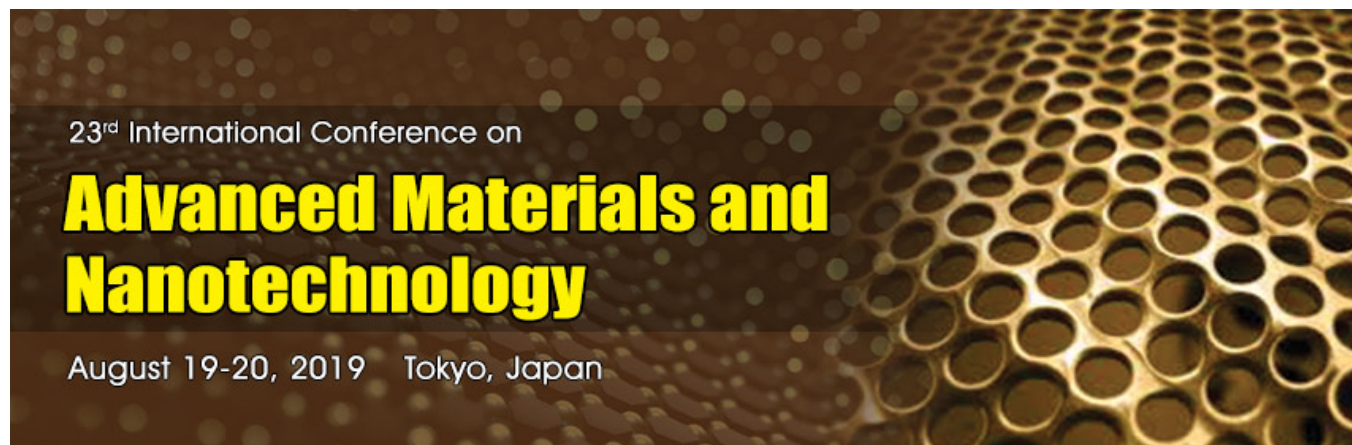
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## MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS



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August 19-20, 2019 Tokyo, Japan

### Theme: Exploring the Possibilities in the Field of Advanced Materials and Nanotechnology

Advanced Materials and Nanotechnology involves the tracks like Advanced Materials and Functional Devices, Engineering Materials, Composite Materials, Magnetism & Multiferroism, Optical materials and plasmonics, Energy and Harvesting Materials, Nanotechnology-Basics to applications, Nanopore science, Nanomedicine, Bio Nanotechnologies, Carbon nanostructures and graphene, Spintronics, Nanoparticle synthesis and applications.

**Contact:** <https://advancedmaterials.conferenceseries.com/>

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