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# Efficient encapsulation of low dimensional particles in thin films to obtain functional coatings



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#### ABSTRACT

The incorporation of nanoparticles into a polymeric matrix to improve the mechanical properties or add new functionalities is a very common practice in nanocomposites materials. However, the organic nature of the common matrices difficults this methodology. In this work, low dimensional particles (LDPs) are introduced in an inorganic silica matrix obtained by the sol-gel method, which provides an appropriate host to encapsulate the LDPs. The step of introducing particles with only one of their dimensions in the nanoscale, into an inorganic matrix is not defined in the literature, therefore, a study of process and efficiency of the LDPs encapsulation is carried out by means of measuring structural properties (SEM, XRD, DTA-TG, particle size) and functionalities (colorimetry and abrasion-chemical resistances) of composites in thin films.

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## 1. Introduction

The incorporation of nanoparticles into polymeric matrix to improve the mechanical properties of the composite or to add a new functionality is a very common practice in the nanocomposites [1,2]. In addition, some techniques have been developed to functionalize these nanoparticles in order to improve the interaction with the matrix, since often, its inorganic nature is not compatible with the matrix in which they are incorporated [2].

The sol-gel process is a well-known wet-chemical route to synthesize oxide materials with high compositional homogeneity [3–5], as silica or titanium oxide. In addition, it is a process that uses low thermal treatment, which does not require vacuum and complex technologies, allowing to reduce the synthesis and sintering temperatures of some materials. Depending on the drying and sintering process, it is also possible to obtain high purity materials with different morphologies and properties [6,7]. At slow evaporation condition, the gel volume is reduced in 5 times, and it is formed a xerogel with a more or less compact structure. But in supercritical conditions (high pressure and wet gel), a highly porous and fragile matrix is formed, with a very low 1% shrinkage [3]. The vitreous matrices obtained by the sol-gel method at low temperatures are good candidates to incorporate nanoparticles since the functional organic molecules are able to be occluded in the open pores

\* Corresponding author. *E-mail address:* esther@icv.csic.es (E. Enríquez). of the network and can interact with the environment, liquid or gaseous, and perform functionalities such as pH indicators, temperature sensors or optical phenomena [8-10]. The versatility of the sol-gel method could provide great amount of applications in the nanotechnology field. In coatings obtained by sol-gel process, nanoparticles can be introduced instead of molecules, so that it can produce not only chemical but also physical interactions. Different works synthesize nanoparticles by the sol-gel process [11–13], but very few focused on directly introducing functional nanoparticles into a sol-gel derived matrix [14–17]. Hence, the nanoparticles introduced are mechanical and thermally protected by the matrix, providing an adequate matrixparticle coupling to achieve an improvement of the composite structure. The procedure is more complex when thin films or coatings are aimed to incorporate functional nanoparticles. It will only be possible when nanoparticle dimensions, or at least one of the nanoparticle dimensions, will be lower or similar than the thickness of the coating.

In this work a study of introducing low dimensional particles (LDPs) with different morphologies in a silica thin film obtained by the sol-gel method is carried out. Silica provides an appropriate inorganic matrix to carry out the preparation of homogenous coatings with LDPs due to its chemical stability and its low temperature ceramic processing which provide good mechanical properties useful for many applications [18,19]. The step of introducing nanoparticles with at least, one of their dimensions in the nanoscale is not defined in the literature, hence, it is not an obvious process, since the nature of the particle surface is a very important parameter due to require compatibility between them and additionally to preserve the

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# Table 1

List of samples prepared to characterize the incorporation of LDPs into the silica matrix.

Sample description	Nomenclature
Silica treated at 500 °C-1 h	Silica 500 °C
Silica $+$ 0.5–5 wt.% AlNPs treated at 500 $^\circ\text{C-1}$ h	SALO.5, SAL1, SAL2, SAL5
Silica $+$ 10–50 wt.% Iriodines® treated at 500 °C-1 h $$	SIR10, SIR20, SIR30, SIR50
Silica $+$ 0.5–5 wt.% CNFs treated at 250 °C-3 h	SCNF0.5, SCNF1, SCNF2, SCNF5
Silica + 20 wt.% Iriodines® + 1 wt.% CNFs treated at 500 °C-1 h	SIR20 + SCNF1
Silica + 20 wt.% Iriodines® + 1 wt.% AlNPs treated at 500 °C-1 h	SIR20 + SAL1
Silica + 20 wt.% Iriodines $\! \mathbb{B} + 1$ wt.% AlNPs + 1 wt.% CNFs treated at 500 °C-1 h	SIR20 + SCNF1 + SAL1

functionality and properties of the additives. For this reason, the structural properties and the functionalities of the LDPs, with different morphologies (polygonal, laminar and fiber), are studied

when incorporated into the silica matrix obtained by the sol-gel method.

#### 2. Experimental procedure

## 2.1. Materials

The reagents used to prepare the silica sol are tetraethyl orthosilicate 99% (TEOS, Sigma Aldrich) as silica precursor, ethanol absolute 99.9% (MERCK) as solvent and deionized water to produce the hydrolysis and hydrochloric acid 37% (MERCK) as catalysts. The ceramic substrates used are tiles that consisted of glazed sanitary ware porcelain [16]. The low dimensional particles (LDPs) used are commercial sheets of mica covered with metal oxides such as titanium oxide, iron oxide and tin oxide (Iriodines®, Merck S.A.),  $\alpha$ -alumina nanoparticles (AlNPs, Vicar S.A.) and carbon nanofibers (CNFs, Grupo Antolin S.A.) with particles of ca. 28 µm, 80 nm and 180 µm in average diameter, respectively. The dispersant used for each LDP was Dolapix (1 wt%) for Iriodines and alumina and Triton X-100 (2,75 wt%) for CNFs.

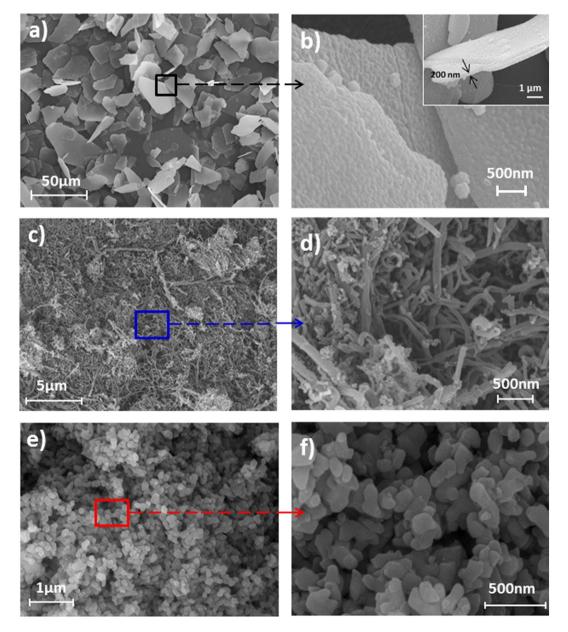


Fig. 1. FESEM micrographs of low dimensional particles at different magnifications: Iriodines® (a and b), CNFs (c and d) and AINPs (e and f).

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