Report on the outcomes of a Short-Term Scientific Mission[1]

Action number: COST Action CA20130

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Details of the STSM

Title: **Coating materials analysis and development for MIC applications**

Start and end date: 01/07/2023 to 14/07/2023

Description of the work carried out during the STSM

First, a visit for the different labs in the department was made, with explanation of which type of equipment they have for which purpose, most of them related to the STSM. For example, the synthesis of chemical composites is made by photochemical approach then, experiments must be performed in a dark room to avoid polymerization before was necessary. Different equipments for microscopy (optical, SEM, etc), equipments for adhesion and mechanical test (LR5K Plus, Lloyd instruments. Ltd., England) and hardness by indentation (Struers, Duramin-40) were explained for what purpose and how to be used in our materials. Also equipment for the polymer and composite synthesis was shown and explain how to use.

We used several nanobiohybrids of copper and copper/silver previously prepared in my lab in Madrid to be tested. Therefore, the development of STSM experimental training part was:

1. *Synthesis of polymer-nanohybrid composite*

For the coating of the metal nanobiohybrids the first step was to prepare the polymer with mix the hybrid for photochemical coating the surface. Different solutions of polymer were tested to finally obtain the best, 60% bisphenol glycerate, 40% TEGAMA acrylate, 0.5% camphor quinone, 1% amine. Curing of the polymer was made with a blue LED light (wavelength range 420-480nm, 5W) lamp, 20s x 3 to ensure the correct preparation. Polymer was well formed.

2. *Carbon steel surface*.

316L Carbon steel pieces were prepared by laser using v= 400 m/s,160W. These pieces were later used for direct adhesion of our synthesized composite.

[1] This report is submitted by the grantee to the Action MC for approval and for claiming payment of the awarded grant. The Grant Awarding Coordinator coordinates the evaluation of this report on behalf of the Action MC and instructs the GH for payment of the Grant.

3. *Test of adhesion of polymer on 316L steel*. (2 step, preparation and secondly attach on the surface)

At this point we want to test the efficiency of the polymer coating on the steel material. For adhesion test we will prepare a polymerized material with 8 mm height x 4 mm. For this we use a special cylindrical mold created by Dr. Furtos with the specific size. For that first we have to put some silicone to allow separate the material after polymerization (3x 20 s lamp). Then when the piece was prepared, it was attached on the surface using polymer liquid as glue. For polymerization we use 3x20 s for 4 times in each direction with lamp horizontal directly to the surface. However, the adhesion of the polymerized material (as blank) was very low being easily separate. This was test in other steel surfaces with similar results.

In order to improve the adhesion, we polish the steel surface but no differences were observed. Conclusion can be that as optical microscope showed, no porosity of the material does not allow a well adhesion of the polymer. At this point in order to improve the porosity for success, we perform sand-blasting on the steel surface. After that, microscope images showed the presence of some porosity on the material. This, after treatment, was washed with distilled water in sonicator for 10 min and then dry at 90 degrees for 15 min. Material after washed showed better performance (more clear black holes). Then steel surface was prepared for adhesion. We repeated simply adhesion of polymer on the treated surface same protocol described above. In this case the piece was strongly attach on the metal surface (by photochemical polymerization), showing a much better performance.

4. *Synthesis of polymer-nanobiohybrid composite.*

Then after solving the surface adhesion, we start to prepare the novel composition mixing polymer liquid and solid nanohybrid. For that, we first evaluate two hybrid concentrations 5%w/w and 2.5% w/w using Cu lyophilized nanohybrid name Cuvirac (cu). For 5% it was used around 200 mg polymer and corresponding solid. Both components were mixed in a glass slide mixing properly until homogeneity. Then this mixer was put in the cylinder mold added carefully to avoid formation bubbles inside. Then 3x20 s light was done and cylinder was opened and piece was perfectly formed as blue (polymer and solid was well mixer). Then the 8 mmx4 mm material, bottom surface was covered with fresh polymer (trying to cover only the 4 mm surface and then located vertical on the treated-steel surface. Photo-treatment was then performed for polymerization with 3x 20 s horizontal in all directions (4 directions W-E-S-N). Procedure was repeated similar preparing the sample with 2.5% cuvirac.

Then next, we selected 2.5% amount for preparation of the other hybrids-polymer mixers. Cu/Ag1% hybrid was prepared by triplicate as previously described 3 times (sample turn blue-greenish color after polymer mixing). A third sample, Cu/Ag hybrid (highest silver content) did not fully polymerized by these conditions (composite sample was completely black against grey color of the solid hybrid). Therefore, in this case first sample was refilled and again 3x20 seconds and second cylindrical sample was prepared using curing 6x20s to ensure polymerization.

Also for future hybrid efficiency as antimicrobial activity and for hardness testing, circle samples of 8 mmx 1 mm were prepared, 2x20s for each face for LED curing.

5. *Evaluation of composite properties*.

Then, different polymer-hybrid composite prepared attached on the steel or directly were tested for mechanical properties and, especially, adhesion capacity.

Description of the STSM main achievements and planned follow-up activities

The results obtained after evaluation properties of the new polymer-hybrid materials showed a very good properties of adhesion. Highest value of adhesion strength was found when the amount of hybrid was lower, for example in composite containing 5% cuvirac, the adhesion strength was 8.23 MPa whereas with 2.5 %hybrid was 9.31 MPa. The presence of silver in the hybrid also had effect reducing the adhesion strength of the material to 5.12 MPa for Cu/Ag1% or 4.12 MPa for Cu:Ag. In any case, all composite prepared show a greater adhesion strength which will be enough for future application as antimicrobial coating materials.

In term of the hardness of the material after introducing hybrid, the Vickers hardness was measure. The Vickers hardness test uses a square-based pyramid diamond indenter with an angle of 136° between the opposite faces at the vertex, which is pressed into the surface of the test piece using a prescribed force, F. The time for the initial application of the force is 2 s to 8 s, and the test force is maintained for 10 s to 15 s. After the force has been removed, the diagonal lengths of the indentation are measured and the arithmetic mean, d, is calculated. The Vickers hardness number, HV, is given by:

HV = Constant × Test force / Surface area of indentation (HV1: 9.81 N)

The hardness of the material was preserved by introducing our hybrid in polymer or even increased in the case of composition with silver, polymer only gave a value of 16.92 HV1/8s while of cu:ag was possible of 21.82 (samples measured in quintuplicate).

Finally, compression capacity as mechanical properties were evaluated. It was shown that hybrid inclusion on the polymer increase the compression, 1.76MPa compared with 1.2MPa of polymer alone. Inclusion of 1% Ag in the hybrid improved the property of the final material with a compression value of 1.9MPa. However, the presence of high quantity of Ag in the hybrid showed almost not affecting to the compression properties (1.1 MPa).

These results show that it was possible to create composite by combining our material as additive in a polymer, as final resin which also can be used to attach it in steel surfaces. These will be of great value for next evaluation of antimicrobial properties, especially focus on application of microbiological corrosion inhibition. The test evaluation demonstrate that the final material showed enough strength to be able to use in hardness conditions. Therefore, we can conclude that the incorporation of our hybrids in polymer matrix could increase adhesion properties, compression and hardness of the material.

Therefore, planned goals after these results will be testing the catalytic efficiency of the novel composite and antimicrobial properties for application against MIC analyses. STSM will allow to discuss to complete the publication of review article in collaboration about materials with antimicrobial properties (materials and techniques used in these STSM), which will be submitted soon. Results obtained in these STSM will be published alone or in combination with other recent results focused on MIC for contribute to specific objectives in this Cost action.

Also this STSM will allow me to obtain interesting knowledge in chemical polymerization processes to applied in my lab and interesting analytical techniques and continues and open novel research collaboration action with this research group in the development of novel coating effect for MIC applications.