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

Action number: CA17107

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Details of the STSM

Title: Smart textiles functionalized with photocatalytic biocarbon for indoor air purification.

Start and end date: 15/03/2022 to 08/04/2022

Description of the work carried out during the STSM

Description of the activities carried out during the STSM. Any deviations from the initial working plan shall also be described in this section.

The tested samples consisted of olive stone-derived activated carbon (OSAC) loaded with different amounts (5 wt% and 10 wt%) of photocatalysts, namely Fe_2O_3 and MnO_2. However, samples prepared with MnO_2 didn't show any activity during the first preliminary tests, so we prepared new samples using TiO_2 to compare with Fe_2O_3 performance at the end.

Samples: Pure OSAC, OSAC- 10 wt% Fe_2O_3, OSAC- 10 wt% TiO_2, OSAC- 5 wt% Fe_2O_3- 5 wt% TiO_2, and one textile impregnated with OSAC- 5% Fe_2O_3 – 5% TiO_2.

Adsorption capacity test: The performance of the different samples for gaseous methyl ethyl ketone (MEK) adsorption was evaluated by a static adsorption experiment. Prior to testing, samples were oven-dried overnight at 70 °C to remove any residual moisture. The test was carried out in a closed glass box with a volume of 18 L equipped with a small electrical fan. A Petri dish containing 0.1 g of sample powder was placed inside the testing chamber on a metallic support, and 1 µl of MEK solution (equivalent to 16 ppm) was injected into the chamber through the inlet using a 10 µl syringe. The fan was turned on to distribute the MEK evenly and homogenise the concentration inside the box. The chamber was hermetically sealed, and the MEK concentration was continuously recorded for one hour using a photo-ionisation detection (PID) sensor connected to the computer, which enabled direct results acquisition using LabVIEW software. The temperature and relative humidity were controlled via sensors during the experiment and were adjusted to 20 °C and 35 % - 45 %, respectively. Three repetitions per substrate were conducted, and samples were regenerated in the oven at 70 °C for 24 h. The experiments were

[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.

performed in dark conditions, and the MEK removal percentage was calculated using the measured MEK residual concentration after one hour in the chamber as follows.

$$MEK\ adsorption\% = \frac{Ci - C1h}{Ci} * 100$$

Where C_i and C_{1h} are the measured MEK concentrations at the start of the experiment and after one hour, respectively.

Integrated adsorption-photocatalytic degradation test:

The same experimental setup of the adsorption capacity test was utilized. A visible light lamp (irradiance = 10 W/m^2) was fixed above the test chamber in a way to effectively irradiate the sample (0.1 g). The experiment started under dark conditions by injection of 1µl of MEK via the inlet, and the change in MEK concentration was recorded by the sensor continuously. When the concentration was stabilized, the sample was considered saturated, and no more adsorption was allowed. The sensor was turned off, and a second injection was done. A 30 min waiting period was allowed with the electrical fan switched on to reach a homogeneous distribution of the pollutant in the chamber. Afterward, the light was switched on to irradiate the sample and activate the photocatalysts, and the evolution of MEK concentration was recorded again for one hour. The integrated adsorption-photo-degradation (IAP) efficiency of MEK was estimated by the following equation:

$$MEK\ IAP\ \% = \frac{Ci - C1h}{Ci} * 100$$

Where C_i was the concentration of MEK after 30 min from the second injection and C_{1h} was the concentration of MEK measured after 1 hour with the light switched on.

The pure photocatalytic agents, as well as OSAC-PCs specimens, were tested.

NOx degradation test: the NOx degradation activity of the pure photocatalytic agents and the photocatalytic carbon hybrids was evaluated according to the method described in UNI 11247 standard. Firstly, 0.1 g of sample powder was mixed with 5 mL of ultra-pure water and ultrasonicated for 5 min. Subsequently, the mixture was evenly spread on a rectangular-shaped glass (10 cm × 5 cm) covered with aluminum foil and dried in the oven at 70 °C. The obtained sheet was put in the testing apparatus, which consisted of a cylindrical glass chamber with a volume equal to 3 L. NO gas was supplied with a flow rate of 1.5 L/min, and the initial concentration was adjusted to be under 500 ppm. The tests were performed at ambient conditions (temperature and relative humidity). NOx and NO rates were measured using a Nitrogen oxides analyzer (Model 8841, Rancon instruments s.p.a). NOx photocatalytic degradation was assessed under visible light, solar light, and a UV light lamp. The irradiance of each lamp was measured using a photo/radiometer (HD 2102.2, Delta Ohm), and the light intensity was adjusted to 10 W/m^2, 1.5 W/m^2, and 17 W/m^2 for visible, solar and UV, respectively.

SEM-EDAX analysis:

The pure and photocatalytic activated carbon samples were characterised by scanning electron microscopy (SEM, Tescan Vega3) coupled with an energy disperse X-ray analyzer (EDAX) to investigate the morphology and elemental composition of the specimens. Images and data were collected using Vega TC software.

Note: After observation of the results from the previous tests, one **textile sample** impregnated with OSAC-5% Fe2O3-5% TiO2 was prepared and tested for NOx removal. The same procedure used for the powder samples was done. However, in this case, the functionalized textile was directly tested without the need of glass support.

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

Description and assessment of whether the STSM achieved its planned goals and expected outcomes, including specific contribution to Action objective and deliverables, or publications resulting from the STSM. Agreed plans for future follow-up collaborations shall also be described in this section.

During the STSM period, we could perform the planned tests despite the challenges that were faced at the beginning, particularly for optimising the test conditions and adjusting the experimental setup, which took additional time. Briefly, the main results are summarized as follows:

Adsorption capacity test: the samples exhibited high capacity towards MEK adsorption under ambient conditions. The adsorption percentages after one hour ranged between 63 % and 78 %. Olive stone-derived activated carbon adsorbed higher MEK quantity compared to the reference commercial activated carbon.

Integrated adsorption photocatalytic degradation (IAP) test:

Results from the IAP capacity for pure photocatalytic agents demonstrated the low photocatalytic ability of the tested photocatalysts under visible light. In fact, the photocatalytic capacity was about 18 % and 21 % for pure TiO_2 and pure Fe_2O_3, respectively. Samples made of OSAC-PCs showed promising results and values were about 65 %, 71 %, and 83 % for OSAC- 10 wt% Fe_2O_3, OSAC- 10 wt% TiO_2, OSAC- 5 wt% Fe_2O_3- 5 wt% TiO_2 respectively.

NOx degradation test: The photocatalytic NOx conversion by pure photocatalytic agents was higher compared to OSAC-PCs mixtures. Pure Fe_2O_3 showed higher activity under visible light conditions, and NOx degradation rates were about 21%, 14%, and 16% under visible, solar, and UVA irradiations, respectively. However, TiO_2 showed better performance under UV conditions, where the NOx degradation rate was up to 38%. Under visible and solar lights, NOx removal rate by TiO_2 was about 6 % and 37 %, respectively.

Interestingly, the sample containing both photocatalysts (OSAC- 5 wt% Fe_2O_3- 5 wt% TiO_2) showed a higher ability toward NOx degradation under solar light compared to the sample containing only one type of photocatalyst, which informs about the potential synergetic effect between Fe_2O_3 and TiO_2.

SEM-EDAX analysis: SEM images showed that the photocatalysts particles were well distributed over the porous structure of the activated carbon. However, some areas revealed the presence of aggregates due to the tendency of photocatalytic nanoparticles to agglomerate, which could be improved by extending the ultrasonication phase applied to the activated carbon-Photocatalysts mixtures during the preparation process. Moreover, the EDAX provided the detailed atomic composition of the samples.

The collected data are intended to be used in writing a scientific paper that covers the topic of the efficiency of photocatalytic activated carbon in air purification under indoor conditions. We also discussed future follow-up analysis of new samples, and we agreed that another visit could be performed in the future to test the photodegradation ability of MEK under solar light and also to evaluate the durability of the samples (change of the photocatalytic performance after continuous testing cycles). In addition, the findings of this STSM can be presented at the next CONTEXT cost action conference.

To sum up, this STSM was a great opportunity and an enriching experience that helped me to gain some practical knowledge about indoor air remediation and to experience using new equipment. Both SIMAU group and Innorenew CoE are open to being involved together in future research work and continuing the collaboration that started with this STSM.