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TREATMENT OF HOLLOW GLASS BUBBLES WITH SILANE COUPLING AGENT. SEM ON HOLLOW GLASS BUBBLES, BEFORE AND AFTER TREATMENT WITH SILANE COUPLING AGENT

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Abstract In this study will be presented, how 20 millilitres of Silane Coupling Agent, adhered on 5 grams of Hollow Glass Bubbles (HGB), how the micro bubbles are looking before and after mixing, how to filter the hollow spheres from the agent and what needed to be taken in consideration when using coupling agents in processes. This paper will show how Silane Coupling Agent(3-Aminopropyl) triethoxysilane, adhered on the Hollow Glass Bubble (HGB). It is expected to observe at HR-SEM (High- Resolution Scanning Electron Microscopy) how the Hollow Glass Bubbles looks when the Silane Coupling Agent (KH-550) is applied on the filler vs when is not. During the process will be concluded, what risks should be taken in consideration, when using Silane Coupling Agents on Hollow Glass Spheres and what important information/ steps are needed to be taken in consideration before and after coupling treatment.

Keywords. HGB, Silane Coupling Agent, HR-SEM, Hollow Glass Spheres, (3-Aminopropyl) triethoxysilane.

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Materials

In this study were used from 3M, Zwijndrecht, Belgium- Glass Bubbles, grade iM16K[1] (density 0.46 g/cm^3) and Silane Coupling Agent (KH-550) supplied by Expert-Trade Romania, from Thermo Fisher Scientific, A10668- (3-Aminopropyl) triethoxysilane, 98%[2].
Hollow glass bubbles or hollow glass spheres, known as low in density, were selected for this experimental study, conducted at University Politehnica of Bucharest: Faculty of Material Science and Engineering Laboratory.

Figure 1: Hollow Glass Bubbles from 3M - Grade iM16K

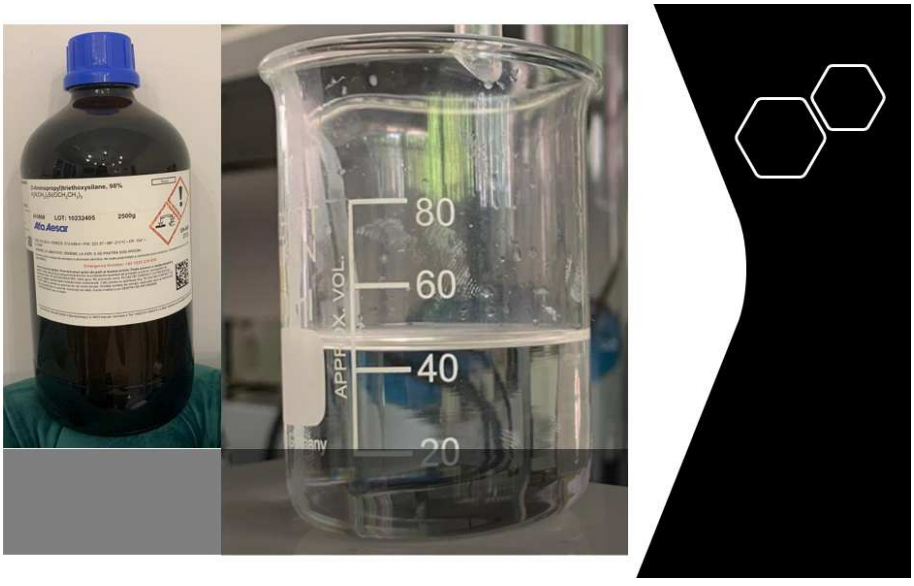

Figure 2: Silane Coupling Agent A10668 (3-Aminopropyl) triethoxysilane, 98%

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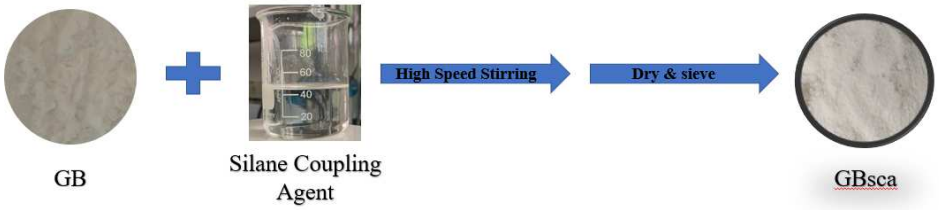

Figure 3: Schematic illustration of preparing procedure for the Glass Bubbles and Silane Coupling Agent

Sample preparation

5 (g) grams of Hollow Glass Bubbles were immersed in 20 (ml) millilitres of Silane Coupling Agent, in a trough glassware and scaled with an Adventurer laboratory model. The powder of hollow glass spheres was stirring by hand for 2 minutes and then the mixture was filtered through a laboratory paper map filter, from Filtrak, with medium wide pores/ medium fast filtering for crystalline deposits, using a laboratory separating glass funnel (Figure 4).

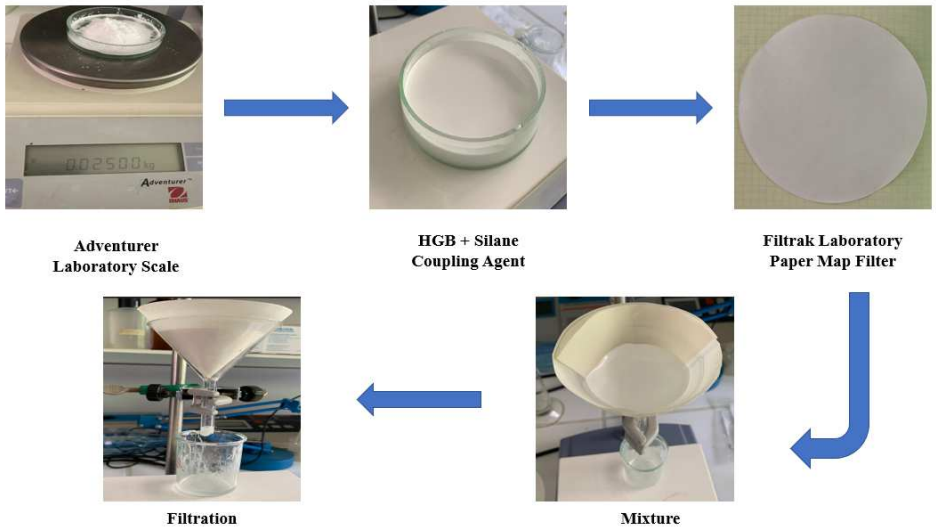

Figure 4: Schematic illustration of preparation, mixture and filtration for the Glass Bubbles and Silane Coupling Agent[3]

The next day, the mixture was dried for 30 minutes, at 65 ˚C in a laboratory thermostat incubator from Calories. After drying in the incubator, was noticed a small top crust, which appeared on top of the mixture (Figure 5). To bring the crust back to powder phase, has been used a Laboratory Mortar, where all the treated mixture was tiny chopped, until the powder phase of the mixture was gained.

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Figure 5: Schematic illustration of drying and mashing the treated Hollow Glass Bubbles[3]

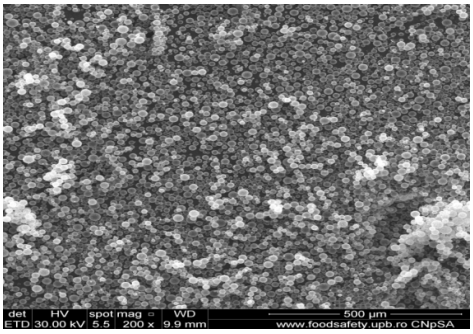
Characterization

The morphology of the treated and untreated Hollow Glass Bubbles was investigated by a High-Resolution Scanning Electron Microscopy (HR-SEM, resolution of 1.2 nm, Material Science and Engineering Laboratory, UPB)

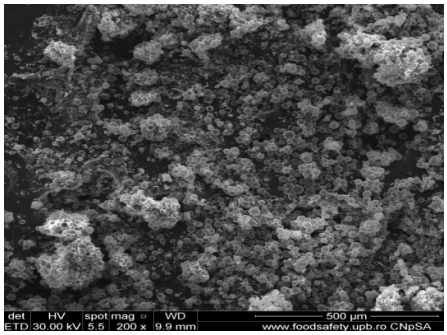
Results and discussion

Morphology

Table 1 shows the morphology of the treated vs untreated with Silane Coupling Agent with the Hollow Glass Bubbles, at different resolution (200x/ 500x/ 1 000x/ 4 000x/ 10 000x magnification).



(a) SEM of HGB at 200x



(a)$_1$SEM of HGB$_{sca}$ at 200x

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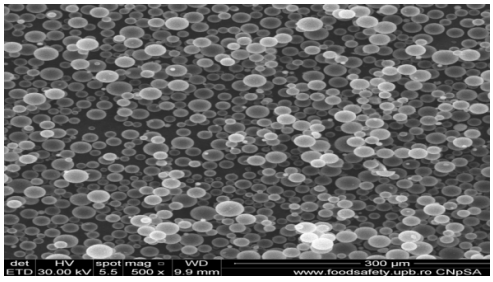
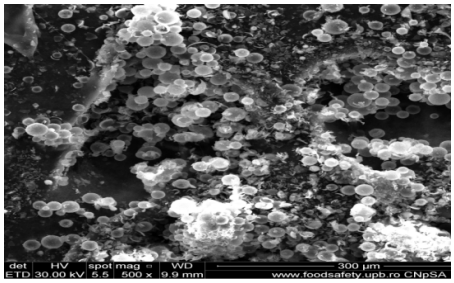
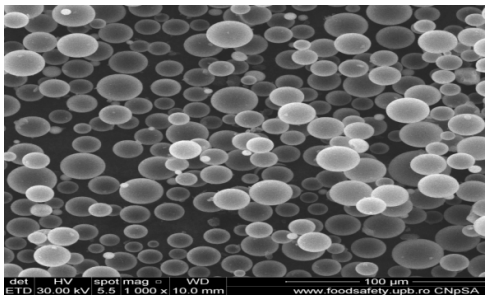


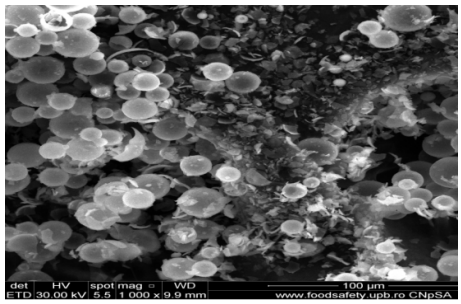
(b) SEM of HGB at 500x



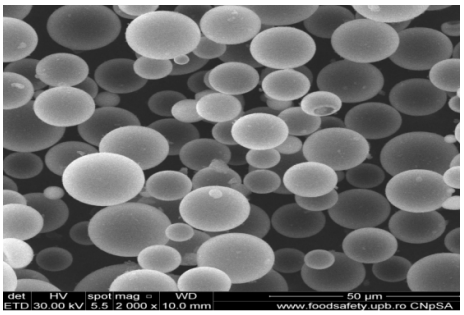
(b)$_1$SEM of HGB$_{sca}$ at 500x



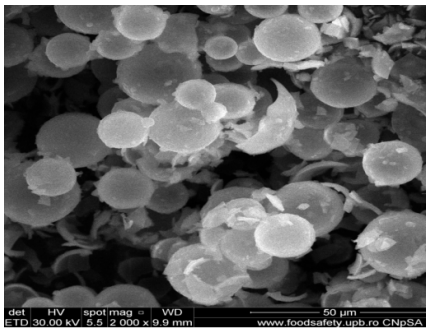
(c) SEM of HGB at 1 000x



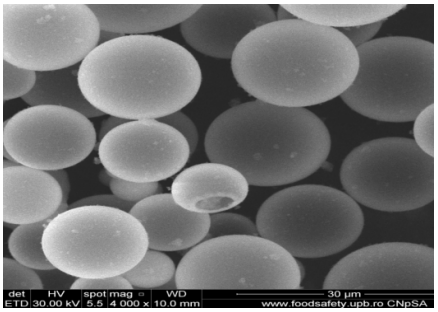
(c)$_1$SEM of HGB$_{sca}$ at 1 000x



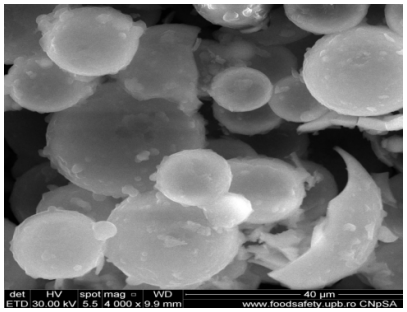
(d) SEM of HGB at 2 000x



(d)$_1$SEM of HGB$_{sca}$ at 2 000x



(e) SEM of HGB at 4 000x



(e)$_1$ SEM of HGB$_{sca}$ at 4 000x

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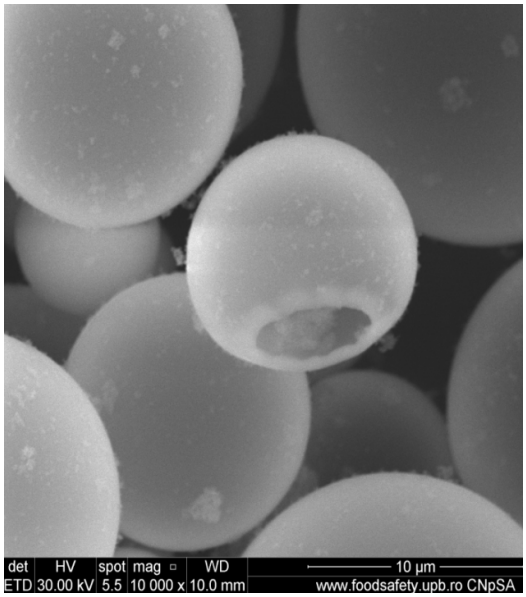
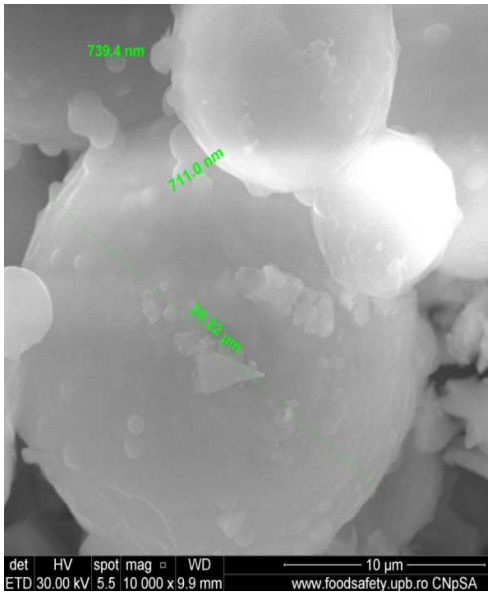


(f) SEM of HGB at 10 000x



$(f)_1$SEM of HGB_{sca} at 10 000x

Table 1: Morphology of the HGB at (a)200x, (b)500x, (c)1 000x, (d)2 000, (e)4 000, (f)10 000x; Difference between untreated HGB (a,b,c,d,e,f) vs treated HGB (a_1,b_1,c_1,d_1,e_1,f_1).[4]

Conclusion

HR-SEM shows how the adhesion between Silane Coupling Agent and Hollow Glass Microspheres adhered in a good shape.
Untreated HGB were broken in some cases Table 1 (d, e, f), most common reason is due to the transportation conditions. Because of the mashed mixture process, which took place in the laboratory mortar, the number broken HGBsca (treated microspheres) are in a bigger number Table 1 (d_1,e_1,f_1). As reference for the next study, will be used a sprayingapplication for the Silane Coupling Agent and a caution way for drying and sieving on the post process applications for the microspheres.

References

[1] 3M Belgium MSDS ae available at *http://www.mmm.com/be,* Accessed: 03/06/2021

[2]Merck, Romania Home, Search Results, A10668- (3-Aminopropyl) triethoxysilane, 98% *https://www.sigmaaldrich.com/catalog,* Accessed: 03/06/2021

[3]Environmental Engineering and Corrosion Laboratory, Polytechnic University of Bucharest, Romania, 2021

[4]Scanning Electronic Microscopy Environmental Engineering and CorrosionLaboratory, Polytechnic University of Bucharest, Romania, 2021