Wood – Ceramic Composites: Synthesis and Analysis of $GdPO_4 \cdot H_2O$:Eu Modified Wood

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Introduction

Wood is one of the most widely used building materials in residential and non-residential buildings, furniture constructions, and decoration. Due to high sustainability, thermal and electrical insulating properties, and aesthetics, the demand for wood is increasing every year. Nevertheless, wood possesses some undesirable intrinsic properties, such as dimensional instability, inferior mechanical strength, flammability and low resistance to biological and weathering factors limiting its usage as an engineering material. Herein, to enhance mechanical and thermal stability, wood can be modified using various materials..

This work proposes inorganic $GdPO_4 \cdot H_2O$:Eu compound produced by in situ hydrothermal syntheses as a possible substitute to various organic and polymeric compounds to functionalize wood products.

Methodology

The starting materials were $NH_4H_2PO_4$ (99,99% trace metals basis, Sigma-Aldrich), $Gd(NO_3)_3 \cdot 6H_2O$ (ACS reagent, ≥99,0%, Sigma-Aldrich), $Eu(NO_3)_3 \cdot 6H_2O$ (ACS reagent, ≥99,0%, Sigma-Aldrich), distilled water and ethanol (CH_3CH_2OH, 96%, Vilniaus degtinė). Wood-ceramic composites are prepared in three stages – first wood specimen is placed in a low vacuum for desorption of air contents from lumen following the 24-hour immersion in $NH_4H_2PO_4$ precursor solution. Swollen specimen (with precursor solution) was placed in Teflon vessel containing gadolinium and europium nitrate solution. Hydrothermal reaction was carried out at 110 °C for 18 hours. After synthesis, the characteristics of the samples were observed by scanning electron microscopy (SEM), computed tomography; optical properties were determined by luminescence analysis. Thermogravimetric (TG) analysis was performed to attest the effects of GdPO4 on the thermal degradation of wood. X-ray diffraction (EDX) analysis was performed to determine the $GdPO_4$ phase.

Results

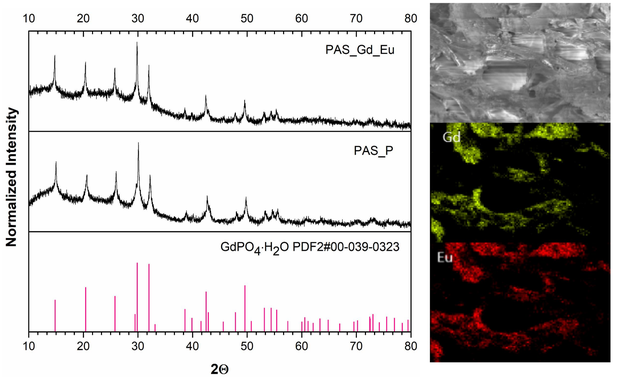

Fig. 1 X-ray diffraction patterns of precipitates after in situ hydrothermal syntheses in wood's matrix: dependency of precursor order. Elemental mapping of the wood surface after in situ hydrothermal $GdPO_4$:Eu synthesis in woods matrix using vacuum pretreatment

Each observed peak matched the standard XRD data. In situ hydrothermal synthesis does not affect wood's mineral part. No additional peaks are presented in diffractograms – wood's hydrolysis does not affect $GdPO_4 \cdot H_2O$:Eu formation.

Due to organic origin, obtained EDX images are of poor quality. Nevertheless, it was found that the surface of the wood is evenly covered with Gd and Eu elements – the elemental distribution corresponds to the morphology of the lumen.

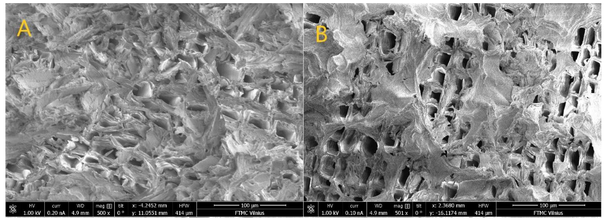

Fig. 2 SEM images of the wood surface after in situ hydrothermal $GdPO_4$:Eu synthesis in woods matrix using vacuum pretreatment: A – 0.03 mol/L, B – 0.06 mol/L

Analyzed samples possess part coverage of $GdPO_4 \cdot H_2O$:Eu sediment. All the cases reveal visible clogging of the pores – cell walls exhibit a thin layer of gadolinium phosphate. Higher surface coverage was observed with increasing precursor concentration, while cell walls coating remained the similar thickness..

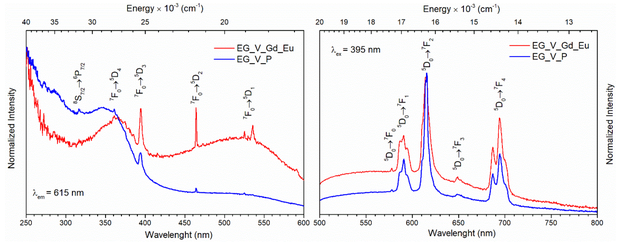

Fig. 3 Excitation and emission spectra of woods matrix end grain (EG) after in situ $GdPO_4$:Eu hydrothermal synthesis

The excitation spectrum of $GdPO_4$:Eu^{3+} in wood's end grain (EG) matrix consists of lines with a maximum at 393 nm between 250 and 600 nm. The lines between 250 and 600 nm arise from f→f transitions within the Eu^{3+} $^4f_6$ electron configuration. The emission spectra of wood end grain samples were recorded under monitoring at 395 nm excitation wavelength, as shown in Figure 18. The emission spectra display several characteristic peaks ranging from 500 to 800 nm, which coincides with the transitions from the excited state $^5D_0$ to the excited ^7F_J (J = 0,1,2,3 and 4) state of Eu^{3+} ions.

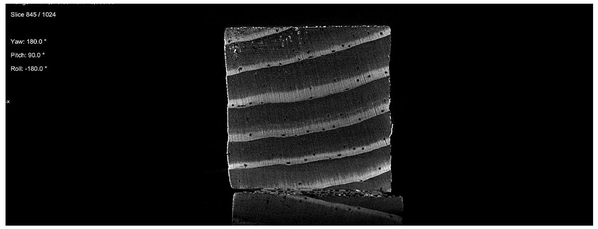

Fig. 4 Radial projection of wood specimen synthesized at precursor concentration six times greater than initial concentration.

Exploring the obtained tomographic image layers, diffusions to deeper layers of the wood specimen can be seen. Diffusion at the left corner as deep as 3 mm is seen in figure 4, starting at 800 slices from 1024 slices. The image reveals that diffusion is more probable to happen from radial orientation (end grain side). Due to open tracheid cells at the end grain $GdPO_4 \cdot H_2O$:Eu particles are more likely to be formed in deeper layers as seen in obtained results.

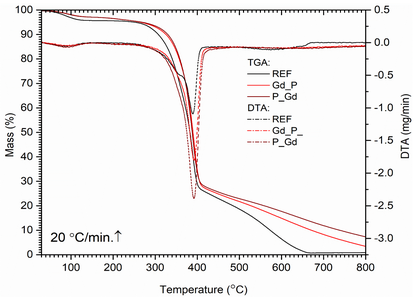

Fig. 5 TG/DTG curves of pure wood and wood treated with $GdPO_4$: Gd_P – initial precursor concentration wood specimen at first stage treated with Gd/EuNO3 solution. P_Gd – initial concentrations wood specimen at first stage treated with $NH_4H_2PO_4$ solution

The first stage shows a weight loss between 3 – 5% to 120 °C, 5% for the untreated wood sample (REF), and 3% for the $GdPO_4$ treated samples. The second stage of weight loss takes place between 280 °C and 410 °C. The mass loss of REF and GP_Eu is the most prominent – reaching 73%. DTA curves show that pyrolysis of treated $GdPO_4 \cdot H_2O$:Eu in wood took place over slightly wider temperature ranges at 330 and 430°C. Samples treated with $GdPO_4 \cdot H_2O$:Eu have a second-stage shift to the higher temperature range. It can be assumed that the combustion process is delayed.

Conclusion

The studies performed suggest that the synthesis of wood-ceramic composites was successfully performed at 110 °C temperature by in situ hydrothermal synthesis. Wood-ceramic composites may possess a significant improvement on retardancy and optical properties. Studies of compounds and methods to prepare composites are still under investigation.