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Study of a Metal-Organic Composite (Wx%Lig) for the Gamma Radiation Attenuation

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Research Article

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ABSTRACT

The objective of this work was to use tungsten and lignin as precursors to produce a metal-organic composite (Wx%Lig) using different sintering processes. Tungsten is a refractory metal and was selected for the composite due to its superior physical and mechanical properties (mechanical strength, high melting point, and excellent cross section with thermal neutrons). Its choice is also justified because it is widely used for high-energy radiation shielding. Lignin extracted from lignocellulosic biomass was selected to be the organic precursor for the composite because it has multiple applications; it is used in the production of aromatics, adhesives, and as a phenolic resin replacement. Analysis of the composite was performed after sintering processes using a Nexview 3D optical surface profiler and analyse the gamma radiation attenuation coefficient using cobalt source (Co-60). Metal-organic composites in ratios of W5%Lig and W2.5%Lig were produced after different heat treatment processes. Then, the gamma attenuation coefficients of the composite samples in these rations were analysing. The gradient of the attenuation coefficient differed when standard tungsten and the composites of W5%Lig and W2.5%Lig were compared with free source Co-60.

INTRODUCTION

Numerous materials are of interest as possible components of new composites due to their wide application in various sectors such as transportation, civil construction, automotive, naval, aerospace, and nuclear. The pulp industry is a significant contributor to the world economy and has shown interest in research and technological development aimed at refining the waste produced by the process of extracting the cellulose fibres contained in the lignin matrix [1,2]. Tungsten is a refractory metal and was chosen to be combined with lignin due to its extensive physical and mechanical properties including mechanical strength, high melting point, and excellent cross section with thermal neutrons (e.g., tungsten is widely used for shielding from high energy radiation) [3,4].

Lignin-extracted lignocellulosic biomass was the organic precursor chosen to be was extracted and used in the samples because of its utility in different types of compounds such as the production of aromatics, adhesives, and as phenolic substitute resin [5,6]. The objective of this work was to create a composite using lignin and tungsten as precursors (in the ratios of W5%Lig and W2.5%Lig) at the different thermal treatment processes and analyse the gamma radiation attenuation coefficient [7]. The techniques used were, analysis Nexview 3D optical profilometry and measurements of the attenuation coefficients of gamma radiation using cobalt source (Co-60). The Nexview 3D analysis of the new metal-organic composites showed different grades of

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surface roughness as a function of the heat treatment process on different concentrations of lignin [8]. Measures of the attenuation coefficient of gamma radiation showed promising results; thus, this new composite may be useful in the transportation of small quantities of highly radioactive substances, a need of the Institute of Energy and Nuclear Research of Brazil [9].

MATERIALS AND METHODS

Processing of the W2.5%Lig and W5%Lig composites

The procedure for producing the composites consisted of preparing homogeneous mixtures of the precursors, tungsten and kraft lignin, in proportions of 9.75:0.25 and 9.5:0.5, respectively. The lignin was obtained from the black liquor of the kraft extraction process of cellulosic pulp [2]. The kraft black liquor lignin was removed and isolated via carbonation, then acidified at pH 2. First, carbonic gas was bubbled into the hot black liquor; between 75 and 80% of the lignin was recovered by filtration. In the second step, the filtrate was treated with sulfuric acid; approximately 10% of the kraft lignin recovered as a powder [6].

Metal samples of powdered tungsten were used as precursors. They were subjected to several levels of granulometry using different sieve numbers (in the order of 200 and 250 μm) in order to form a composite with a higher degree of homogeneity [7,8]. To prepare the samples, the precursors were weighed according to the desired mass ratios for the composition of the composites—in stoichiometric mass proportions from 2.5% lignin to 97.5% tungsten and 5% lignin to 95% tungsten. Subsequently, the mixtures were homogenised by subjecting them to pressing in a 15-ton press, which produced samples 1.2 cm in diameter and 0.8 cm in height **(Figures 1 and 2)**.

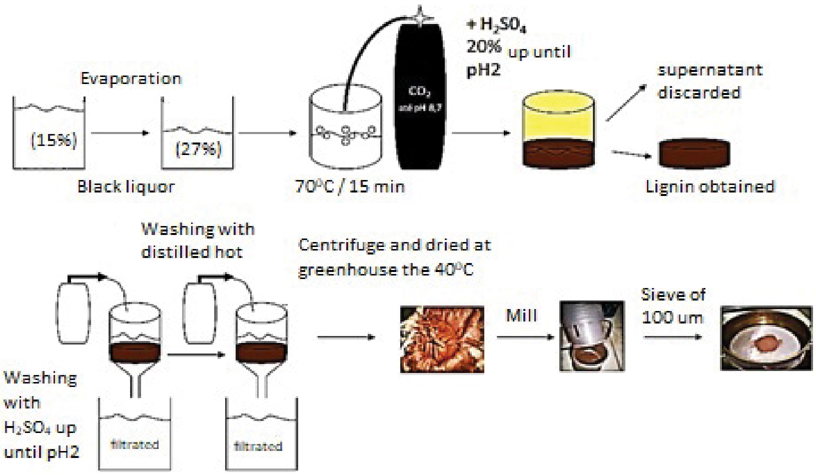

Figure 1. Experimental scheme of the kraft lignin

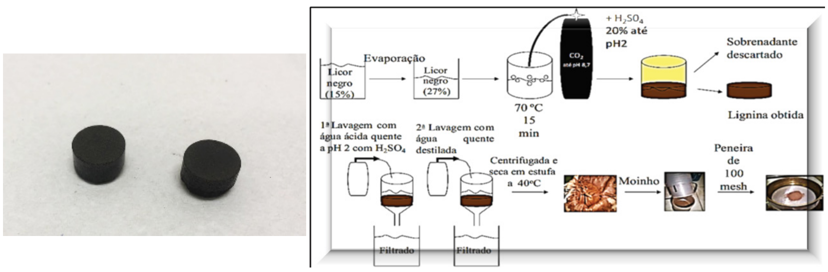

Figure 2. Shows the samples of W2.5%Lig and W5%Lig after pressing

Heat treatment process

The samples were subjected to vacuum heat treatment in a quartz tube; they were heated at a rate of 10 °C min^{-1} at 90 and 100°C for 1 h at each temperature. **Figure 2** shows the samples after pressing and heating. Figure 3 depicts the Edwards mechanical vacuum pump that was run at a pressure of 1.5 x 10^{-1} Torr.

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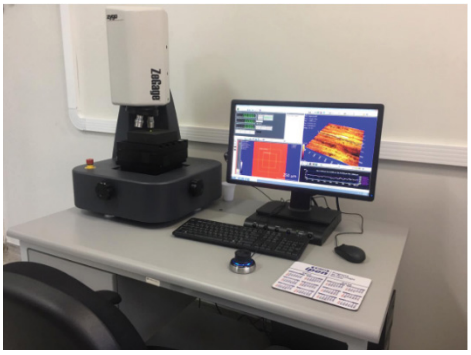

Figure 3. The vacuum heat treatment system

Nexview™ 3D optical surface profiler

After the heat treatment, a Nexview 3D optical surface profiler was used to measure the surfaces of the samples with sub-nanometric precision and independent of the field of view. Software Mx software was used for analysis; it allows complete system control and data analysis, including interactive 3D maps, quantitative topographic information, intuitive measurement navigation, and embedded SPC with statistics, control charts, and limits. This software was used to determine the density and porosity of the samples (**Figure 4**) [10].

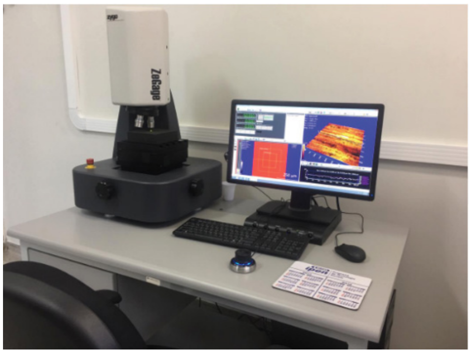

Figure 4. The Nexview ™ 3D Optical Surface Profiler

Measurements of the gamma radiation attenuation coefficient

The experimental apparatus for measuring the gamma radiation attenuation was set up in the laboratory of Physics/USP and consisted of the following items:

- Cobalt-emitting source, with two characteristic peaks at 1130 and 1330 keV.
- Source containing seven cobalt pellets (Co-60) inside a lead vessel.
- A GW INSTEK Oscilloscope (GDS-2062).
- Spectroscopic amplifier with a High Voltage Stabilizer Source (mod. TCH 1500-2E).
- XCOM software to compute the 0.140 MeV energy of gamma rays emitted by the decay of Mo-99, installed for storing the photons count.
- Gamma emission detector - MOD. 2M/2 BICRON ASSEMBLED IN INDIA (Figure 5).

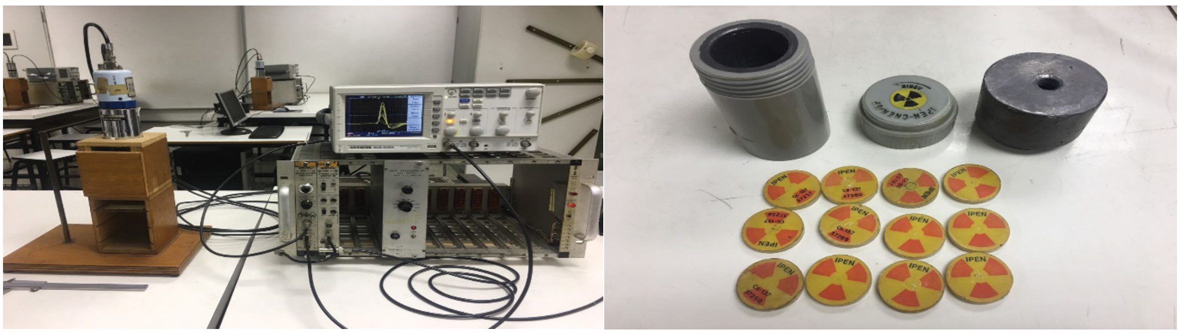

Figure 5. a) Experimental scheme measuring gamma radiation; b) Source cobalt (Co-60)

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RESULTS AND DISCUSSION

The results from the Nexview 3D optical surface profiler are shown in Figures 6 and 7; the degrees of roughness and porosity of the W5%Lig samples after heat treatments at 90°C and 100°C in a limited area of 250 µm are depicted. The sample treated at 90°C had more pores than the sample treated at 100°C, this difference can be attributed to the increased temperature and melting of the lignin; it functioned as an adhesive between the metallic tungsten grains, decreasing the composite's roughness.

Figures 8 and 9 show the degrees of roughness and porosity of the W2.5%Lig samples after heat treatments at 90°C and 100°C in a limited area of 250 µm. The sample treated at 90°C had more pores than the sample treated at 100°C. This result likely means that the sample subjected to 100°C heat treatment underwent greater lignin melting; consequently, the voids between the metallic tungsten grains were more completely filled, resulting in lower roughness. Therefore, the composite samples with less lignin (W2.5%Lig) had higher porosity than the W5%Lig composite samples, a result attributable the concentration and to the lignin's adhesive process and the differences in physical properties of precursors (density). Alternatively, this result can be attributed to the fact that lignin is organic and has adhesive properties and tungsten has higher hardness and larger particles than lignin.

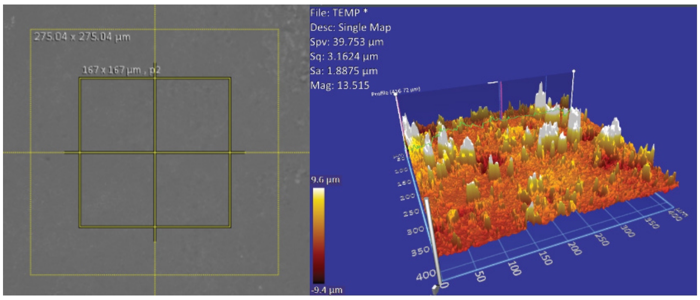

Figure 6. The optical profile of the W5%Lig sample after heat treatment at 90°C

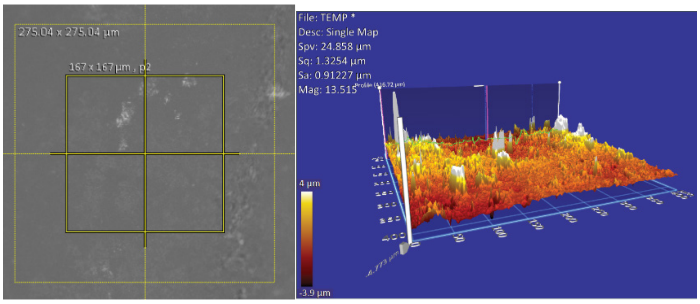

Figure 7. The optical profile of the W5%Lig sample after heat treatment at 100°C

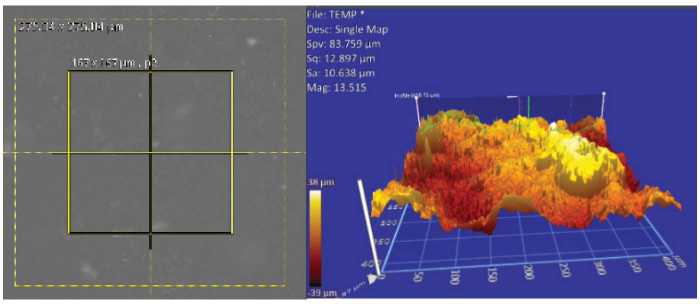

Figure 8. The optical profile of the W2.5%Lig sample after heat treatment at 90°C

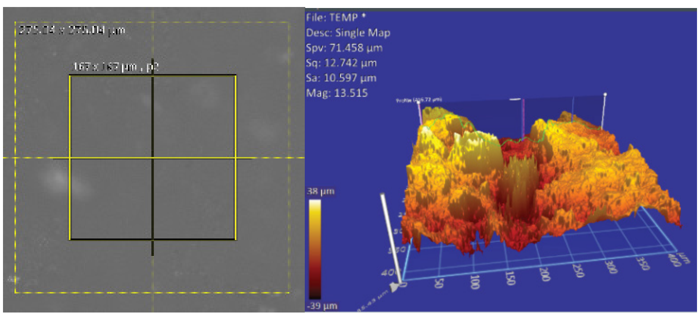

Figure 9. The optical profile of the W2.5%Lig sample after heat treatment at 100°C

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Attenuation of gamma radiation

Figure 10 compares the attenuation of gamma radiation spectrums between a free source of Co-60 and the samples of standard tungsten and composite W5%Lig sintered at 90°C. The results showed that at the two energy peaks characteristic of Co-60, a gradient in the attenuation coefficient around 32% between the free radiation with no obstacles and standard tungsten and around 42% between the free radiation with no obstacle and the W5%Lig composite sintered at 90°C. Thus, the attenuation coefficient of the W5%Lig compound was higher than that of standard tungsten. This result may be attributable to the concentration of lignin present in the composite. The presence of lignin decreases the sample's pore concentration as temperature increases; diffusion of the lignin occurs in the granular insets of the tungsten powder, increasing the cross section and justifying the higher attenuation coefficient [9].

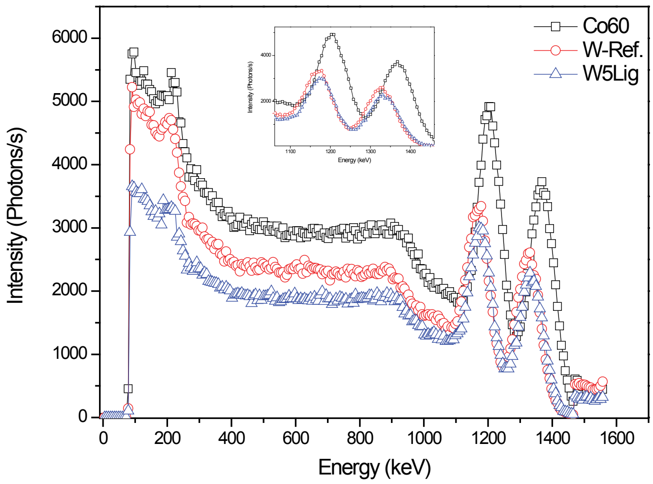

Figure 10. The comparative spectrums of the attenuation of gamma radiation between Co-60, standard tungsten, and W5%Lig sintered at 90°C

Figure 11 represents the comparative spectrums of the attenuation of gamma radiation between the samples of standard tungsten and the W5%Lig composite at different temperatures of sinterization. This outcome may be attributable to the degradation of the lignin as the temperature increased, as it is playing its adhesive role, closing the pores of the composite and approaching the coefficient of tungsten itself, in the other direction, we observe that the lignin shows a good material attenuation in its natural form [10].

Figure 12 represents the comparative spectrums of the attenuation of gamma radiation between the samples of standard tungsten and the W2.5%Lig composite. The results show that changing the lignin concentration in the composite from 2.5% to 5% after the sintering process at 90°C did not cause a significant difference in the attenuation coefficient compared to standard tungsten. This outcome shows that an even larger amount of lignin is needed so that a more significant difference in the attenuation coefficient can be achieved [9,10].

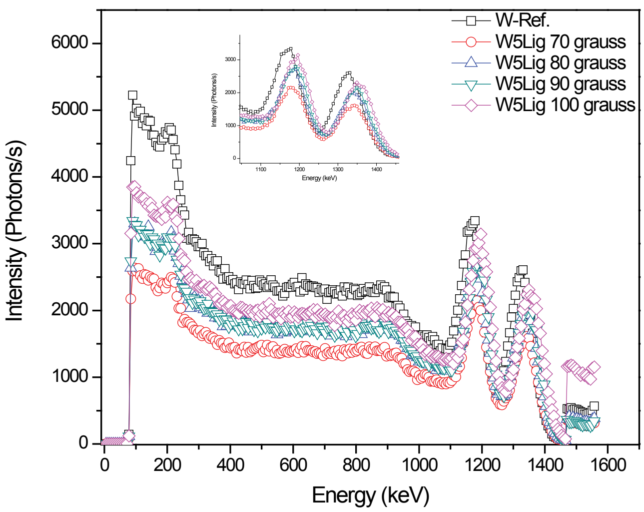

Figure 11. The comparative spectrums of the attenuation of gamma radiation between standard tungsten and the W5%Lig composite sintered at 70, 80, 90, and 100°C

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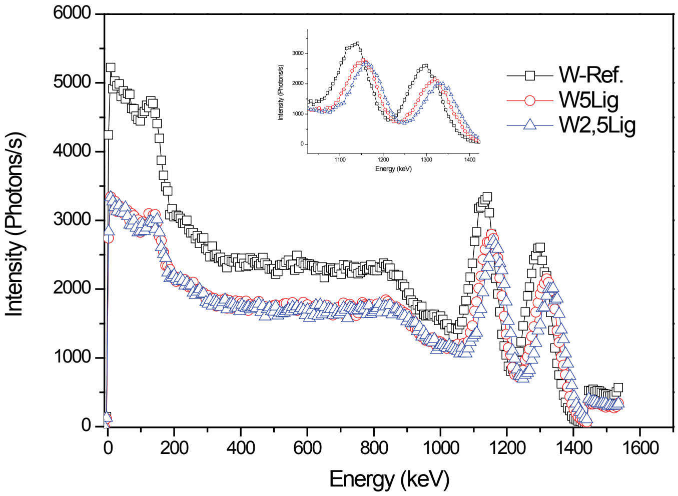

Figure 12. The comparative spectrums of the attenuation of gamma radiation between standard tungsten and the W5%Lig and W2.5%Lig composites sintered at 90°C

CONCLUSION

- The W2.5%Lig and W5%Lig composite samples showed good homogeneity considering that lignin is an organic substance and tungsten has a high affinity for oxygen.
- Optical profilometry analysis showed that increasing the temperature contributed to the agglutination of the tungsten particles, minimising the pore density of the samples.
- The attenuation coefficient between free radiation with no obstacles and standard tungsten showed a gradient of around 32% at the two energy peaks characteristic of Co-60.
- The attenuation coefficient between free radiation with no obstacles and the W5%Lig composite showed a gradient of around 42% at the two energy peaks characteristic of Co-60.
- The attenuation coefficients of the peaks of the W5%Lig and W2.5%Lig composites were not significantly different.
- The gamma attenuation coefficient of the kraft lignin increased as temperature decreased.
- The results showed that kraft lignin represents an excellent option with which to form a tungsten composite, but additional research is required. For example, the gamma attenuation coefficient using pure lignin and different concentrations of tungsten remains to be determined in next paper.

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