



Synthesis of crosslinked films based of 3-(trimethoxysilyl) propyl methacrylate silanized xanthan gum/lignin and their crosslinking by gamma radiation, for potential application as films packing

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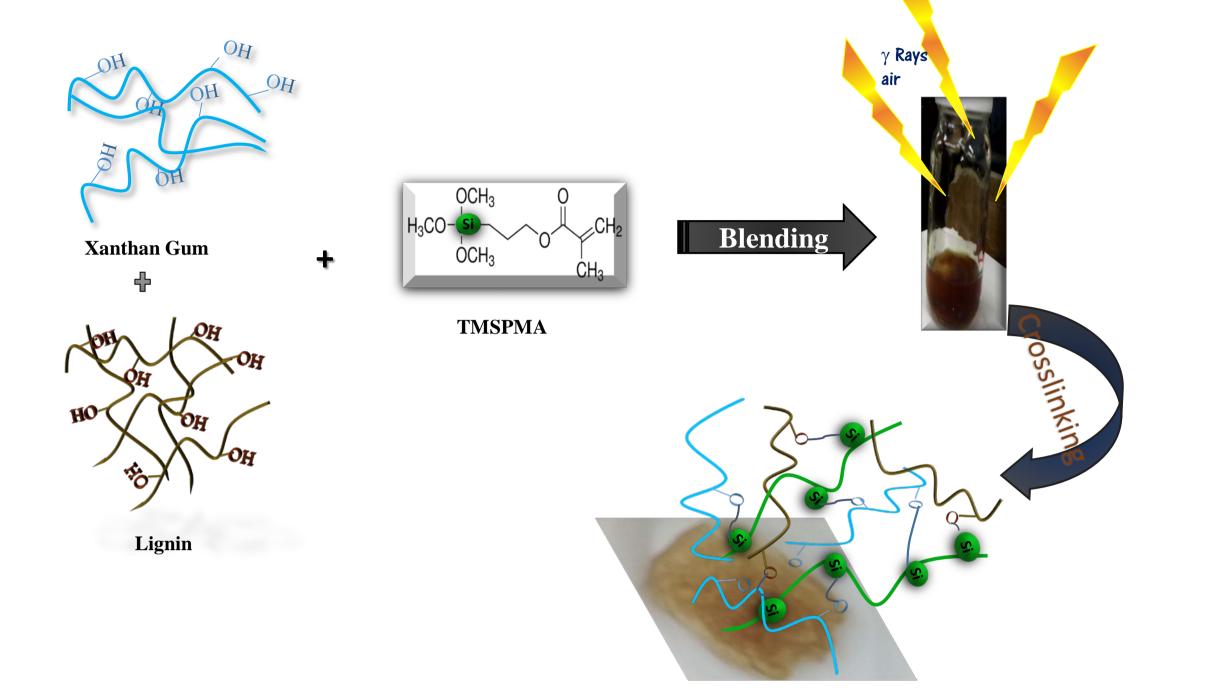
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INTRODUCTION

The production of biodegradable and edible films based on biopolymers has attracted attention and represents one of the most advanced challenges in the field of food packaging and coating. Polymers derived from natural products offer the greatest opportunities as component of edible films since their biodegradability and environmental friendly [1]. Xanthan gum has been used in a wide variety of foods, for a number of important reasons, including emulsion stabilization, temperature stability, compatibility with food ingredients and its pseudoplastic rheological properties [2]. Xanthan gum is classified E 415 in the European List of Permitted Food Additives, so xanthan gum is recognized as a non-toxic additive for human consumption. Lignin, a natural biopolymer, mostly derived from wood, is an enormous and renewable reservoir of latent polymeric materials and aromatic chemicals. Due to their very complex structure, lignins are amorphous polymers with rather limited industrial use. They are usually seen as waste products of pulp and paper industry and often used as fuel for the energy balance of the pulping process [3]. Unfortunately, the use of biopolymers as food packaging materials has drawbacks such as poorer mechanical, thermal, and barrier properties as compared to the conventional nonbiodegradable materials made from petroleum. The incorporation of nanofillers such as silicate, clay, and titanium dioxide (TiO₂) to biopolymers may improve not only the biopolymers' mechanical and barrier properties but also offer other functions and applications in food packaging such as antimicrobial agent, biosensor, and oxygen scavenger [4]. Several physical an chemical treatments are employed to incorporated the inorganics compound to biopolymers, among them the gamma radiation, which offer several advantages mainly minimum time requirement, any organic solvents and curing at ambient temperatures. In this work, the xanthan gum/lignin mixture was silanized and crosslinking with 3-(trimethoxysilyl) propyl methacrylate by gamma radiation, in order to obtain a material with better properties, for potential application as a packaging film.

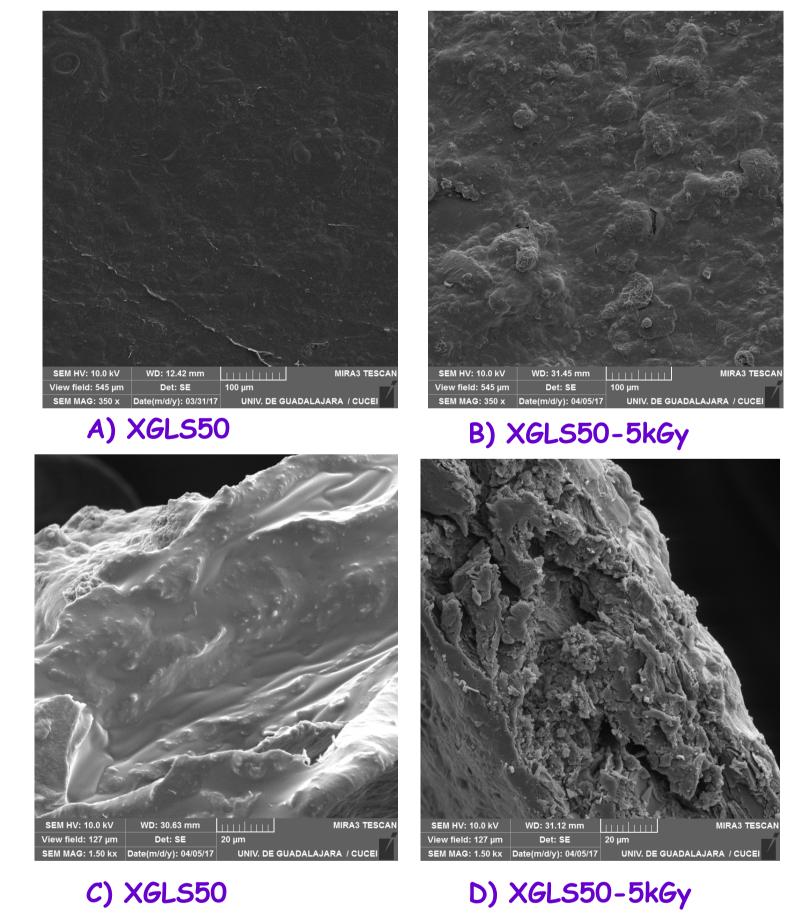




Scheme 1. General procedure to obtain xanthan gum/LS hybrid film. Briefly, a mixture of Xanthan gum (XG) and Lignosulfonate (LS) at 1 % w/v was prepared, then the 3-(trimethoxysilyl) propyl methacrylate (TMSPMA) was added, the mixture was stirring to homogenized. Films were then cast by applying the film-forming solution onto Teflon dishes and allowed to dry for 48 h, at room temperature and at 35% relative humidity. The films were irradiated with gamma rays under air atmosphere at 2.5 y 5 kGy of dose and dose rate of 6.25 kGy/h.







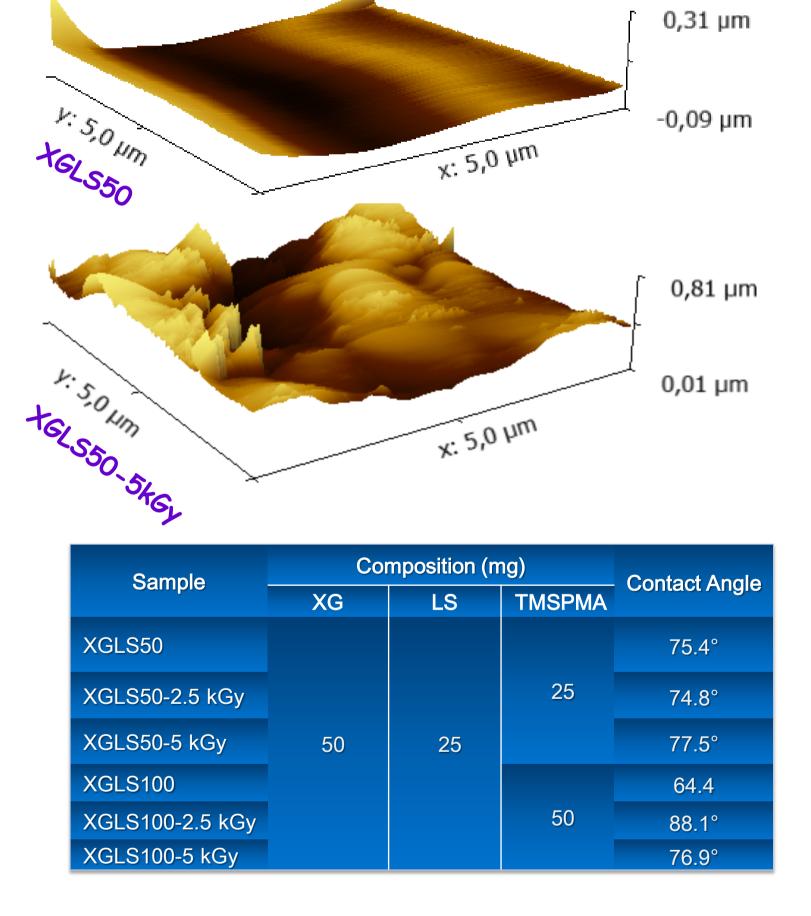
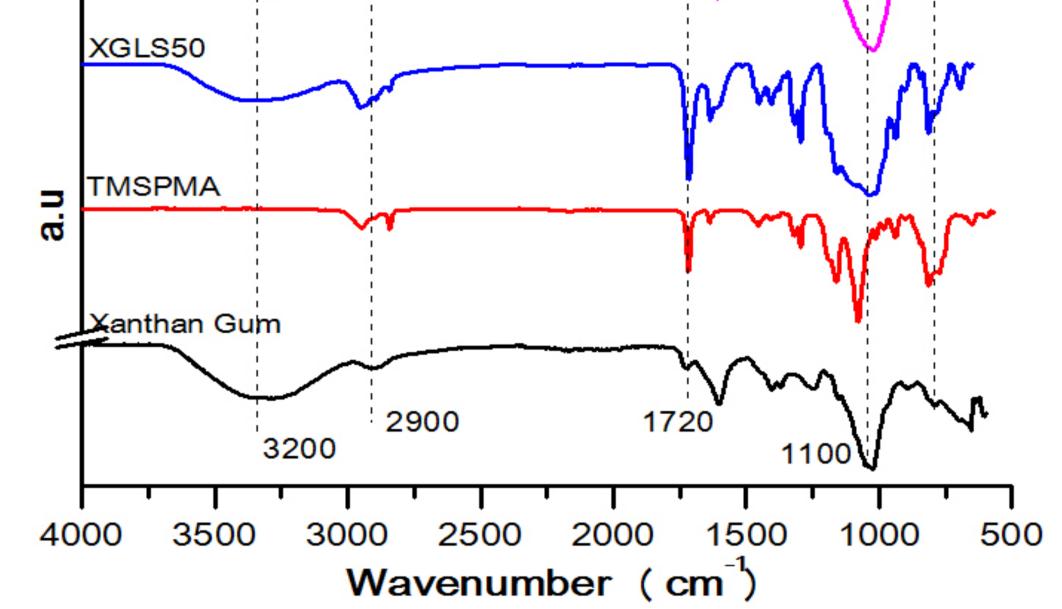


Figure 1. Left: SEM micrographics of surface (A and B) and cross-section (C and D); Right: AFM topography and contact angle quantification of irradiated and unirradiated samples

The chemical composition of film hybrid sample was analyzed by FTIR spectroscopy, the spectrum of the film irradiated (XGLS50-5kGy) shown than the bands at 1720 and 3200 cm⁻¹ correspond to carbonyl and hydroxyl group respectively increased. While that the bands at 2900 and 1100 cm⁻¹ correspond to -CH2- and XG glucoside group respectively are broad, due to the addition of -O-C=O, -Si-O-Si- and -Si-OH groups of TMSPMA, also appear the new band at 800 cm⁻¹ correspond to -Si-O group. The thermogravimetric analysis (TGA) shown that the samples silanized and crosslinking with gamma radiation enhanced their thermal stability, the XGLS50-2.5 kGy and XGLS-100 2.5 kGy started to loss mass at 240 °C and 220 °C respectively, while that the XG and LS started at 218 and 180 °C respectively. Additionally, all films had a residual mass (representing char content) at 650 °C, and this was higher when films were silanized and crosslinking. The surface SEM image of the unirradiated films (A) displayed a homogenous and softness morphology compared with the roughness and granular surface of irradiated film (B), due to that the alkoxysilanes with gamma radiation could be formed the porous particles. The cross-section of the irradiated (D) film showed a rough and porous appearance, while the XGLS50 (unirradiated) was observed a compact and slightly roughness surface. On the other hand, the AFM topography shown similar behavior, the unirradiated samples displayed smooth surface, while irradiated film looks irregular and roughness. On the other hand, the contact angle results shown that the hydrophilicity of films treated with gamma radiation is similar to unirradiated films, due that to the roughness and porosity increased, which helped to the absorption of water. Nevertheless, when the silane quantity is higher the hydrophilicity decreased.



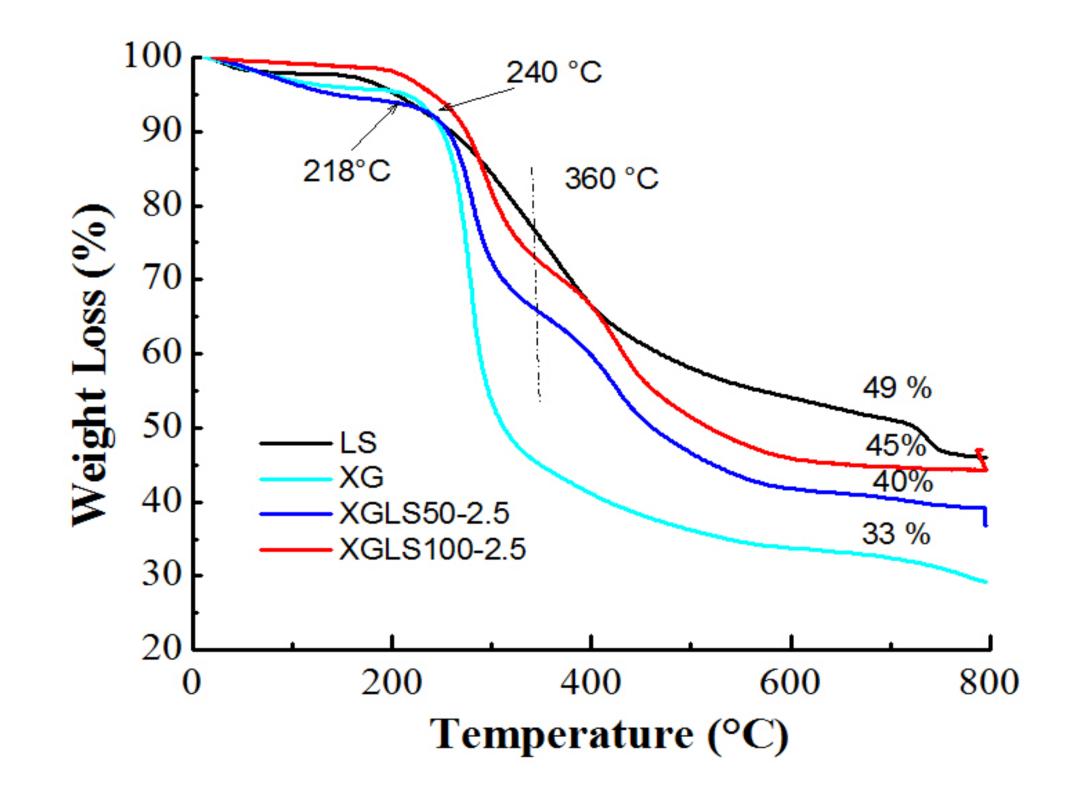


Figure 2. FTIR-ATR spectrum (above) and TGA analysis (below) of different samples.

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