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Hemp Fibres Modification by sol-gel Method for Polyolefin Composite Filling

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Abstract

In this research hemp fibres have been modified by the sol-gel method in different combinations to implement silica nanolevel coating on fibres surface without compromising the mechanical properties of the fibres and ensure sustainable coverage to achieve matrix-filler compatibility in composite as filler. The coatings of samples prepared using different proportions of nano-sols. The fibres were immersed in the prepared sols made of ethanol, tetraethyl orthosilicate (TEOS), distilled water and hydrofluoric acid. Scanning electron microscopy (SEM) has been used to examine the nature of the surface modification. Energy dispersive X-ray (EDX) analysis was used for chemical composition analysis on coated fibres surface. Analyses based on the SEM show that small sol components proportion differences significantly affect coating adhesion.

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Keywords: sol-gel method, hemp fibers, composite filler, silica coating

1. Introduction

The sol-gel processes are not widely used at the industrial level for coatings purposes. However, the recent evolution of the organosilanes market may open the door to accurately designed materials created under mild conditions. Emerging application for organosilane chemistry is the development of “smart” surfaces that are uncharacteristically hydrophobic or hydrophilic. Improved hydrophobic effects of natural fibres materials and more

hydrophilic surfaces of synthetic fibres can be obtained through the use of sol-gel technologies. The use of octamethylcyclotetrasiloxane and an atmospheric glow discharge process to the treatment for cotton fabric results in a super-hydrophobic surface (angle $>150^\circ$) and hydrophilic treatment of PET and PP with tetramethylcyclotetrasiloxane are illustrated in literature [1].

An advantage of the sol-gel method is the possibility to obtain thin layers of coating on various materials. The modification method of natural fibres materials show that deposited layers can cover all cotton fabric fibres with enough high adhesion [2, 3]. Depending on fibres final use various surface modification treatment variants could be created. In these processes the parameters of sol synthesis and deposition technology for natural fibres surface modification have to be aligned to avoid fibres damages by improper sol composition and disparity of post-processing temperature to the fibres type and applied sol components.

B. Unger et.al. used TEOS, ethanol and hydrofluoric acid (HF) sol for wood modification to improve dimensional stability [4]. Silica sol and silica sol modified with the zinc acetate dihydrate were used for veneer surface modification to improve water resistance [3].

Traditional silane coupling combined with TEOS, methanol and hydrofluoric acid sol for wood sawdust modification were used to improve composite mechanical and tribological properties [5]. Processing of sol-gel coating in some sources starts with surface clearing in NaOH solution and surface modification used hybrid sols [7, 8]. Using sol-gel method for hemp fibres modification in LLDPE composite matrix can increase filler content up to 50 wt%, fluidity and improve water resistance [9, 10].

2. Materials and methods

Synthesis of silica layer by sol-gel method in this research included the use of TEOS, HF, distilled water and ethanol (Table 1). The main goal in this stage is to find proper proportions between TEOS and HF, as fluorsilanes are important components of hydrophobic film deposited on natural fibre surface.

The hemp fibres used were obtained from a trial plot at the Agriculture Science Centre of Latgale in Vilani district, Latvia, in 2014. Hemp fibres of the variety 'Bialobrzeskie' in growing process were cultivated with active nitrogen fertilizer (N60). The harvested hemp stems were left for dew retting on the field 4 weeks. The hemp residues used were supplied by the factory "Zalers".

Table 1. Materials

Reagents		Manufacturer
TEOS	$C_8H_{20}O_4Si$	Alfa Aesar, German
Ethanol	C_2H_5OH	ES (SIA "Enola")
Hydrofluoric acid	HF	ES (SIA "Enola")

Water hydrolyses the TEOS for polymerization, ethanol used as a co-solvent that get TEOS and water into the same phase for reaction. HF acid as a catalyst helps to make the reactions go faster.

Table 2. Nanosol variants, composition and post-processing temperature

Sol variants	Concentration of TEOS, M	Concentration of HF, M	Drying, $^\circ C$	Heating, $^\circ C$	Heating time, min
a	0.09	0.8	90	120	10
b	0.13	0.8	90	120	10
c ¹	0.14	1.6	90	120	10
c ²	0.14	1.6	90	90	30

¹90 $^\circ C$ drying and 120 $^\circ C$ heating, ²90 $^\circ C$ drying and heating

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