CELPREG

PREIMPREGNOWANE LAMINATY NA BAZIE WŁÓKIEN LNIANYCH DO PRZETWARZANIA W WARUNKACH NISKIEGO CIŚNIENIA

PREIMPREGNATED FLAX FIBRE LAMINATE FOR PROCESSING UNDER LOW PRESSURE CONDITIONS

PARTNERZY PROJEKTU

Instytut Włókien Naturalnych i Roślin Zielarskich

Fraunhofer IMWS

Narodowe Centrum Badań i Rozwoju

Projekt nr CORNET/24/Celpreg/3/2017 współfinansowany przez **Narodowe Centrum Badań i Rozwoju** w ramach programu **CORNET**

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Wkład własny: 33 432 zł

Beneficjent Projektu:
Izba Bawełny w Gdyni
Gdynia Cotton Association
Wykonawca Badań:
Instytut Włókien Naturalnych i Roślin Zielarskich
Institute of Natural Fibres and Medicinal Plants

PROJECT OBJECTIVE

The project concerns the development of preimpregnated laminates reinforced with flax fiber products for the needs of lightweight structural constructions. The project will develop the structures of linear and flat flax fiber products with parameters that ensure the correct process of the prepreg formation and the high quality of composites.

The aim of the research on composite reinforcement was to obtain fabric with structure ensuring minimalization of final product porosity.

In order to increase the adhesion of the fibers to the natural epoxy resin and reduce flammability, flax products will undergo chemical modification processes. Laminates reinforced with flax products will be produced through a cost-effective process of preimpregnation and formation of prepregs under low pressure conditions, using a natural epoxy resin.

PHASING OF WORK AND REASEARCH

I Selection of flax fiber with properties corresponding to the requirements which should be met by the laminates

Fibers from few flax varieties were chosen for the project: Artemida, Luna, Sara, Modran, and a Belgian origin fiber. The flax fiber was tested for its usability in reinforcing epoxy resin structures. Basing on the results of metrological analysis as well as physical and chemical tests, **the Belgian original fiber** *(Figures 1-3)* was chosen to make roving to be applied as reinforcement of laminates.

SHEET OF THE FLAX FIBRE CHOSEN FOR WORKS OF THE PROJECT

FLAX FIBER OF BELGIAN ORIGIN

The fiber is long, with uniform, natural color, properly retted, without the smell of volatile fatty acids. The fiber has characteristic, delicate, soft touch typical of dew retted fiber. The raw material is of uniform linear mass (with low coefficient of linear mass variation), well separated, with a visible scanty number of impurities, without damaged fiber. Among all tested flax varieties, this fiber is characterized with the highest value of mean length, the highest tenacity, the lowest surface energy, the highest thermal stability, and the highest moisture absorption from the air.

BELGIAN FIBER

Source: project documentation

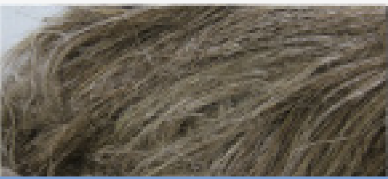


Figure 1. Flax fiber.

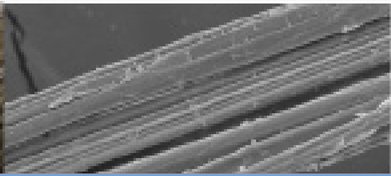


Figure 2. Longitudinal view of flax fiber.

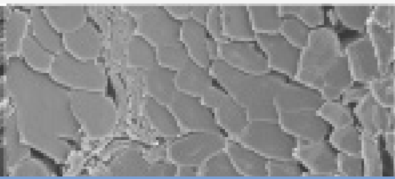


Figure 3. Cross-section view of flax fiber.

Development of fabric structure ensuring low porosity of composite.

The selected fibers were used to spin low twist yarn of linear density 68 tex and natural roving with linear density 400 tex and 2000 tex as well as bleached roving - 2000 tex (Figure 4).

a b c

Figure 4. Low-twist yarn of the linear mass of 68 tex (a), rovings of the linear masses of: 400 tex (b) and 2000 tex (c) natural and bleached.

Physical-mechanical and chemical analyses of rovings and results of trials of roving use as reinforcement to laminate forming allowed to select the following materials: the natural and bleached ones of the linear mass of 2000 tex - for fabric weaving in next stage of the research (Figure 5).

The selected roving had the lowest twist number and the fabric woven with use of the roving were characterised by parallelly laid fibers.

a b

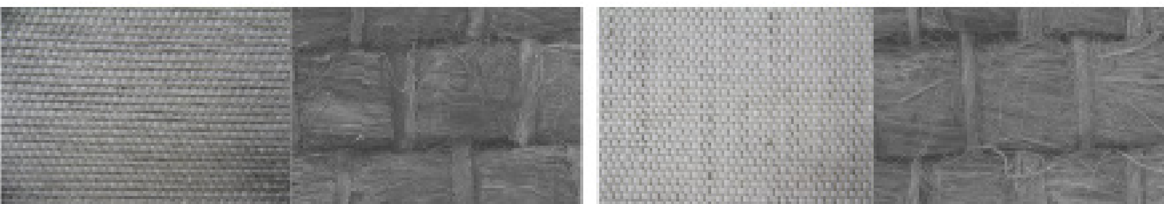


Figure 5. Flax fabrics with the plain weave, made from the 68 tex low-twist yarn (warp) and from natural (a) and bleached (b) rovings of the linear mass of 2000 tex (weft), and microscopic images of fabrics – under the scanning electronic microscope with changing vacuum mode.

The developed fabrics were subject for assessment of their structural, surface and sorption parameters. The fabrics were characterized by high cover factor.

III Modification of selected rovings and textiles: parameters, composition, properties

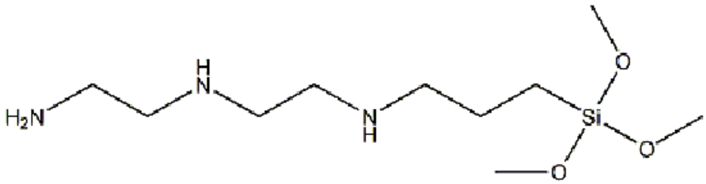
The process of chemical modification of flax fiber products using the silanization method has been developed, in particular for:

- the composition of the modifying mixture
- the parameters of modification process

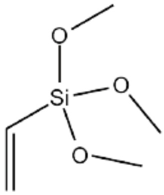
The aim of the modification process was:

- reduction of the flammability of rovings and fabrics made of flax fibers,
- improvement of the properties of products obtained in subsequent stages of the project (fabric, composite)
- improvement of the adhesion with the project's newly developed natural epoxy resins made of linseed.

Two silanes with different structures were selected: amine and vinyl (Figure 6). They are characterized by both, the best compatibility with epoxy resin, as well as the greatest reduction in flammability of modified linen fibers.



3- (DIETHYLTRIAMINE) PROPYL TRIMETHOXYSILANE



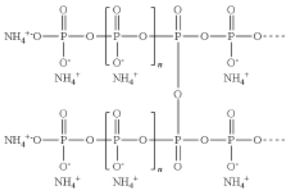
VINYL TRIMETHOXYSILANE

Figure 6. Silanes selected for the project.

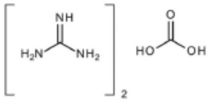
The modification process was carried out in accordance to defined parameters, such as silane concentration, temperature, pressure, time.

Both, rovings and prepared fabrics, were modified. The most advantageous results were obtained in the case of modification of fabrics and those were selected for conducting the subsequent research. The modification in this case was more homogeneous, and thus effective.

In order to achieve the expected reduction in flammability, **four flame retardant compounds were selected** (Figure 7). The process with their addition was carried out in many variants, testing the obtained products both, in terms of reduced flammability and compatibility with the developed epoxy resin (p. IV).

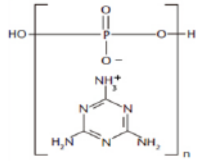


AMMONIUM POLYPHOSPHATE



GUANIDINE CARBONATE

PENTAERYTHRIT



MELAMINE POLYPHOSPHATE

Figure 7. Selected flame retardant compounds.

The modification of the fabric developed on the basis of a raw roving with a linear mass of 2000 tex with the addition of vinyl silane at a low concentration and the addition of ammonium polyphosphate **turned out to be the most advantageous.**

Figure 8 below shows the results from the microcalorimeter (PCFC) comparing the flammability of the fabric modified with vinyl silane and the silane with the addition of ammonium polyphosphate. A **spectacular reduction in the flammability** of the fabric was achieved.

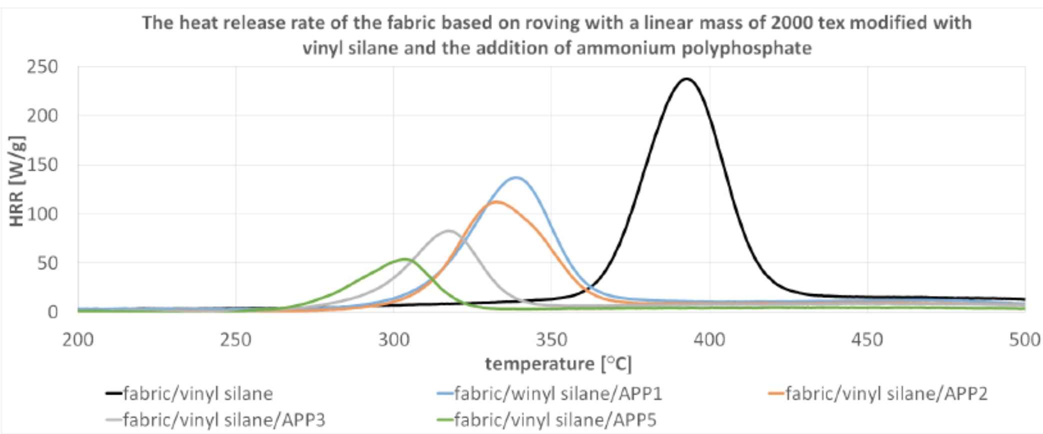


Figure 8. Curves of the heat release rate of the fabric based on roving with a linear mass of 2000 tex modified with vinyl silane and the addition of various amounts of ammonium polyphosphate.

IV Linseed based epoxy – its important parameters and modifications.

Synthetic epoxies based on petrochemicals are well known. They have many useful properties, such as high tensile strength and modulus, good adhesion and insulation properties, and excellent resistance to chemical corrosion.

Native epoxies based on fatty acid esters are available in a small scale. Both types of resins can show mutagenic properties, however primary epoxides (obtained from petrochemical products) always show mutagenic properties, while native epoxies, e.g. fatty acid esters from *Vernonia galamensis,* do not.

It should be also noted that the yield of linseed raw materials such as *Lallemantia Iberica* and *Linum isitatissimum* is approximately 750 kg / ha of oil.

An epoxy resin based on linseed oil was selected for research in the project. Various modifications of the resin system were investigated. The increase of the glass transition temperature by over 12% was determined by the method of Dynamic Scanning Calorimetry. According to DIN ISO 306, a 13% increase in Vicat softening temperature was also determined.

Figure 9. Epoxy resin based on linseed. It consists of: modified epoxy, hardener, flame retardant.

V Composite reinforced with flax fabric

Basing on determined surface energy of modified fibers and epoxy system, the suitability of flame retardants was investigated. By focusing, on the one hand, on the appropriate impregnation of the fibers and, on the other hand, on the reduced flammability of the resulting laminates, the productive combination of flame retardant and resin modification was found.

The porosity of the laminates processed under low pressure conditions was reduced and the UL94 HB flammability results indicated that it is a self-extinguishing laminate. The resulting natural fiber laminates showed E-modulus up to 10.000 MPa.

A so-called "prepregs" in the form of a layer of natural fiber fabric coated with a modified epoxy system were tested for storability. A shelf life of 6 months was verified at -20 °C of the produced test laminates under low pressure conditions and a curing temperature of around 120 °C.

Figure 10. Sandwich construction with decking layer made out of natural fibre composite from bleached flax-fibre and linseed based epoxy.

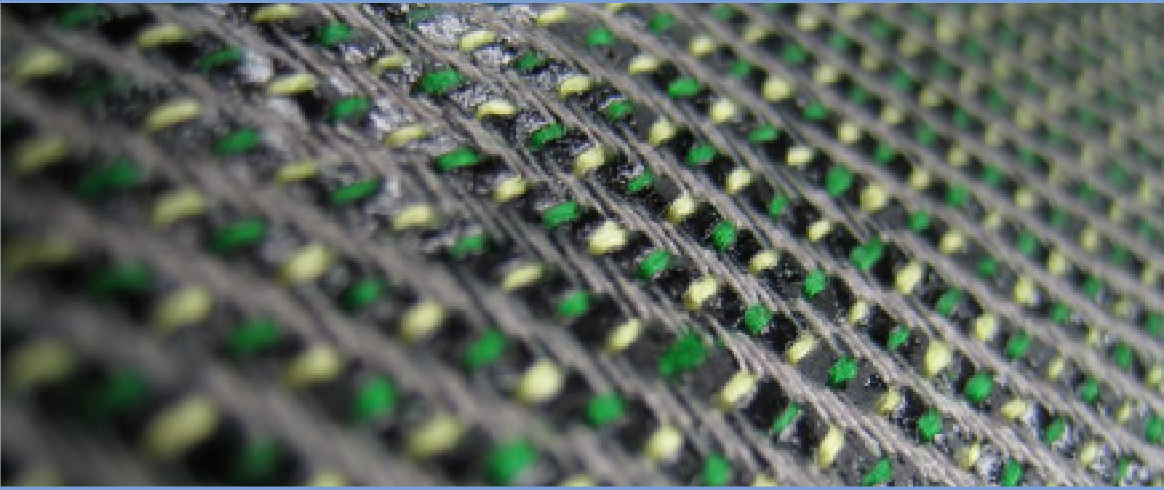


Figure 11. Natural fibers as the visible structure of the laminate. This is especially usable for dyed fibers.

VI Summary

Finally developed composite shows high flame retardancy in a material system with high content of compounds from renewable resources.

By adjusting type and concentration of flame retardant compounds and resin modification to the needs of processing, good compatibility between fibre and matrix was achieved. In result of optimized fibre impregnation in low pressure pressed and cured laminates porosity was reduced.

Tested shelf time of pre-impregnated natural fibres was comparable to conventional prepreg-systems and promises the industrial use of the developed composite.

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