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ANALYSIS AND OPTIMISATION OF REINFORCING MATERIALS FOR WOOD ADHESIVES

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Abstract. Adhesives are very important in the field of glued timber elements. In recent years, more and more attention has been paid to innovations in the field of adhesives for wood materials. Not only in terms of mechanical properties but also in terms of environmental aspects. Wood remains a popular material in the construction industry, but the challenge remains how to ensure the long-term stability and durability of its glued joints. The designed elements must withstand various changes of moisture as well as elevated temperatures throughout the lifetime of the structure. Optimising wood adhesives through appropriate fillers or fibres selection is a step towards more sustainable building materials. The aim of the work was to select suitable fillers or fibres for adhesives based on optimisation through the pairwise comparison method. According to the results obtained, glass (99.98 %) and carbon (95.92 %) fibres can be considered as the most suitable reinforcement material. Basalt fibres, nanocellulose and montmorillonite particles achieved an average of 56 %. TiO_2, SiO_2 and Al_2O_3 particles can be considered as unsuitable fillers for adhesives based on the weight of the selected criteria.

Keywords: Reinforcing materials, fillers and fibres, optimisation process, Saaty's method.

1. Introduction

In the aim of improving the mechanical properties, moisture resistance or thermal stability of bonded joints, various particles are often used as reinforcing fillers and fibres. These particles can contribute to better adhesion of the adhesive to different surfaces, which is a major factor in the automotive industry, construction and other areas [1–4]. The most common particle types include mineral fillers such as SiO_2, $CaCO_3$. These particles can improve hardness and heat resistance or increase the stiffness and workability of the adhesive [1, 2]. There are also organic fillers such as cellulose fibres. Cellulose is the most widespread and important biopolymer on Earth. Nanocellulose has very good physical, mechanical, biological and also chemical properties. It has low toxicity, high strengths, good biocompatibility and extensive possibilities for chemical modification. Nanocellulose can be divided into three types of materials: (I) cellulose nanocrystals-CNC/NCC, (II) cellulose nanofibrils-CNF, (III) bacterial cellulose-BC. Cellulose nanocrystals are obtained by chemical means with dimensions of 3–20 nm (width) and 50–500 nm (length), and by mechanical means nanocellulose fibres with dimensions of 5–100 nm (width), 500 nm to micrometres (length) are obtained [5].

Reinforcing fibres also provide specific advantages. The most commonly used are glass fibres (inorganic origin). They are valued for their high strength, resistance to high temperatures and good chemical resistance. They have a silicate base and are produced by melting 'glass mats' (a mixture of 70 % quartz sand, limestone, potash and colemanite) [6]. The fibre diameter varies by 5–25 µm and the fibres need to be lubricated (this prevents breakage and abrasion of the fibres). For bonding components in the automotive or aerospace industry, carbon fibres are used for specialised applications due to their exceptional tensile strength, low density, corrosion resistance and high temperature resistance. Carbon fibres have been known in technical terms for about 50 years. Basalt fibres also have high strength and resistance to water and chemicals (salty solutions and acids) and elevated temperatures. Compared to other fibres, they have health and environmental advantages [4, 7–9].

In the last few years, the use of different types of particles and fibres, even in nano dimensions, has become a leading form of modification of existing materials, whether in the electrical, textile, food, pharmaceutical and chemical industries, or in the modification of the properties of building materials [10–14]. Nanoparticles can be created by human activity, but also synthetic. Two methods are usually used in the synthetic creation of nanomaterials. The first is a top-down process in which nano-objects are created by gradually reducing the structure of an existing material. The second method is the reverse bottom-up process, where individual atoms or molecules are used to make the resulting nano-objects [15]. The term 'nanomaterial' means a material that is at least 50 % composed of particles with a size between 1 nm and 100 nm [16].

TiO_2 particles (size 250–300 nm) have frequent industrial applications, they are used as additives in varnishes, coatings, paints and plastics. However, the production of particles below 100 nm is technologically and financially demanding, therefore the use of these nanoparticles is only a fraction of the total use of TiO_2 particles. Nano TiO_2 particles have a high specific

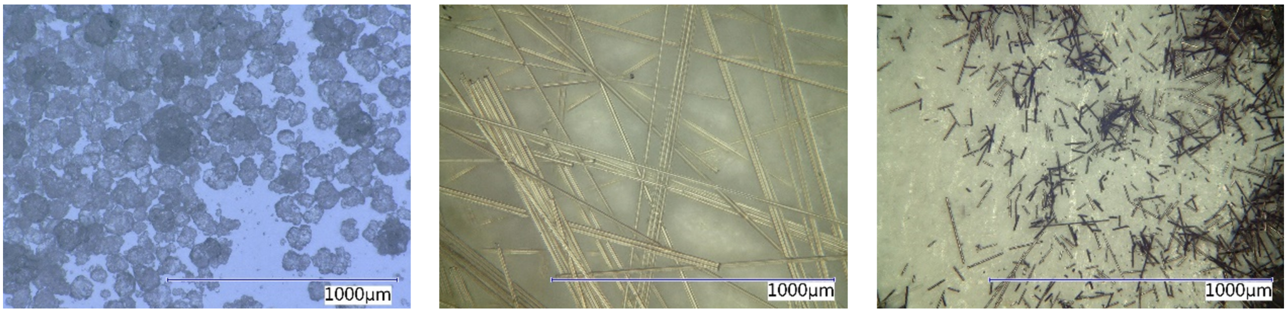

Figure 1. Images of selected filler and fibres as examples of possibilities to modify adhesives. From left: Al_2O_3 powder (magnification 200×), chopped basalt fibres and milled carbon powder (magnification 250×).

Option	**Filler/fibres**	**Availability**	**Price [€ kg $^{-1}$]**	**Size [nm]**
A	TiO_2	Trunnanno (CHN)/Precheza (CZE)	12–80	30–50
B	SiO_2	Trunnanno (CHN)	27–70	10–15
C	Nanocellulose	Sigma Aldrich (USA)	1–9	10–20
D	Al_2O_3	Easchem (CHN)	50–90	20–30
E	Glass fibres	Sklocement Beneš (CZE)	1–2	5 000–25 000
F	Carbon fibres	Sigma Aldrich (USA)	20–25	5 000–15 000
G	Basalt fibres	Kamenny Vek (RUS)	4–5	6 000–21 000
H	Montmorillonite	Sedlecký kaolin (CZE)	20–22	200–500

Table 1. Selected variants of fillers and fibres with parameters for the optimisation process.

surface area but well dispersed in plastics [11].

The best-known clay mineral is montmorillonite, which is most commonly used as a nanofiller. It has a layered, lamellar structure and a large surface area. Clays with montmorillonite as the main mineral are called bentonites. It has many uses, for example in foundry, construction, pharmaceuticals and the cosmetics industry. It is available and cheap [14, 17].

The choice of the right reinforcement material depends on whether the main objective is to improve mechanical properties, reduce costs, or add additional properties such as chemical resistance or thermal stability. The optimum filling dosage for nanoparticle adhesives is around 2 wt. %, for fibres this dosage is slightly higher at around 10 wt. % depending on the type of filler [2, 10–12]. The dispersion of fillers and fibres, which depends on their properties, is a very important parameter in the actual application of adhesives [18]. Fillers and fibres can have high surface tension, and this is the cause of the formation of clumps (agglomerates), which have a negative impact on the mechanical properties of the resulting bonded joints [10, 11, 17]. In order to eliminate the formation of clumps, fillers and fibres are often coated for better dispersion [19]. The adhesive modification process is also dependent on the ecotoxicity of the selected fillers and fibres. The chemicals of concern are assessed using the hazard data obtained and various estimates of the extent to which an organism may be exposed to these substances. These data and estimates are dealt with by the OECD (Organisation for Economic Cooperation and Development), an organisation originating in America. In an effort to use fillers and fibres for routine modification, it is also necessary to consider the price and availability of the selected fillers and fibres. In this work, different fillers and fibres were selected for the optimisation process based on the pairwise comparison method. The aim was to find the optimal fillers or fibres that could have the potential to fulfil the requirements in the field of glued timber joints.

2. Material and methods

Based on research of possible fillers and fibres for structural wood adhesives, four particle-type fillers (TiO_2, SiO_2, Al_2O_3 and montmorillonite clay particles) and four fibre types (glass, carbon and basalt fibres and nanocellulose) were selected. Images from a Keyence VHX-950F optical microscope of possible filler and fibres are shown in Figure 1.

Table 1 lists variants with parameters such as availability, price and dimensions of fillers and fibres. The prices are obtained from freely available databases, they are not prices based on manufacturers/importers offers. The final price depends on the dimensions, the quantity ordered and the degree of purity.

2.1. Optimization process

The correct design of the evaluation criteria is a very important aspect for the selection of the appropriate fillers or fibres. The criteria were chosen based on current environmental trends as well as economic and mechanical requirements. These selected criteria are presented in Table 2.

Each assessment criterion has a unit or has been assigned a number based on a rating scale (Table 3).

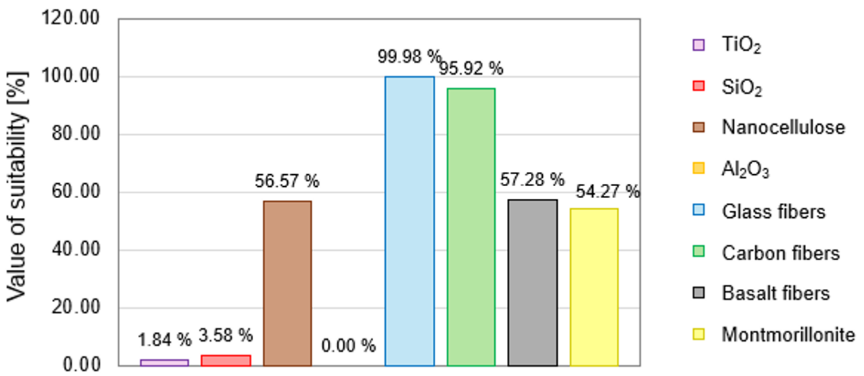

Figure 2. Graphical representation of the calculation matrix.

Number	Criterion	Unit	Rating
1	Workability (dispersion)	[–]	[1–3]
2	Ecotoxicity	[–]	[1–3]
3	Price (p)	[€ kg^{-1}]	[–]
4	Availability	[–]	[1–3]
5	Technological complexity of filler treatment	[–]	[1–3]
6	Max. particle size	[–]	[1–3]

Table 2. Selected criteria to optimise reinforcing fillers and fibres.

Number	A	B	C	D	E	F	G	H	MIN	MAX
1	1	1	2	1	3	3	2	2	1	3
2	2	2	1	2	2	1	1	1	1	2
3	80	70	9	90	2	25	5	22	2	90
4	2	1	1	1	3	3	3	3	1	3
5	3	3	3	3	2	1	1	1	1	3
6	3	2	2	1	3	3	2	2	1	3

Table 3. Evaluation criteria and their values for each option.

The decision matrix (Table 4) consists of a choice of an optimum for each criterion (maximum or minimum value of the criterion). The decision matrix is one of the steps of Saaty's method. It is a pairwise comparison method using multi-criteria evaluation. For this evaluation, the following preference scale was established to calculate the weights of the selected criteria: 1 = equivalence, 3 = weak preference, 5 = strong preference, 7 = very strong preference and 9 = absolute preference.

To determine the weights, the Table 4 was created with the main diagonal containing the units, and values were entered in the remaining cells based on the selected preference scale (element in the row versus element in the column). In the case of a preference for the element in the column at the expense of the element in the row, the inverted preference value was entered into the table cell. The geometric means Si of the individual rows were then calculated. After that the resulting weights Fi of the elements in the rows were calculated. To verify the correctness of the calculation, the sum of the resulting weights must be equal to 1.

After calculating the resulting weights of each criterion, the values of b_{ij} (1) were calculated according to the selected optimum for each filler or fibre depending on the criterion. The optimisation process was concluded by calculating the values of suitability c_{ij} (2), shown graphically in percentages in Figure 2.

$$b_{ij} = \begin{cases} MAX \rightarrow b_{ij} = \frac{a_{ij} - MIN(a_i)}{MAX(a_i) - MIN(a_i)} \\ MIN \rightarrow b_{ij} = \frac{MAX(a_i) - a_{ij}}{MAX(a_i) - MIN(a_i)} \end{cases} \quad (1)$$

$$c_{ij} = F_i b_{ij} \quad (2)$$

3. Results and discussion

The findings show that, when using the same amount of reinforcing material, processability and dispersion are more challenging with nano-sized fillers compared to fibres and larger particles in the millimetre range. The ecotoxicity assessment depends on the dimensions, as small particles are expected to have a more negative impact on the environment than larger particles and fibres. The optimum was chosen as the maximum value for processability, availability and size of reinforcing particles to evaluate the criteria. For ecotoxicity, cost and technological complexity of the filler treatment, the aim is to achieve the lowest possible value. The detailed structure of the evaluation of each criterion is given in Table 3.

The radar chart (Figure 3) presents multidimensional comparison of the essential characteristics of selected filler and fibres for adhesives. By using scaled values and capturing parameters (A-H), this representation enables direct comparison of diverse materials in terms of both economic efficiency and sustainability. For instance, nanocellulose stands out in environmental aspects and availability, while glass fibres demonstrate high dispersibility, which is advantageous for certain industrial applications. This visualized data can serve for informed decision-making when the fillers and fibres are selected, based on specific requirements and technical criteria. To enable clearer interpretation and comparison across selected properties, the

Number	Optimum	1	2	3	4	5	6	S_i	F_i
1	max	1	3	3	9	5	7	472.500000	0.84297090
2	min	1/3	1	1/5	7	5	1/3	0.12962963	0.00023127
3	min	1/3	5	1	9	7	5	87.5000000	0.15610572
4	max	1/9	1/7	1/9	1	1/7	1/9	0.00000467	0.00000001
5	min	1/5	1/5	1/7	7	1	1/3	0.00222222	0.00000396
6	max	1/9	3	1/5	9	3	1	0.38571429	0.00068814
			SUM						**1**

TABLE 4. Saaty decision matrix.

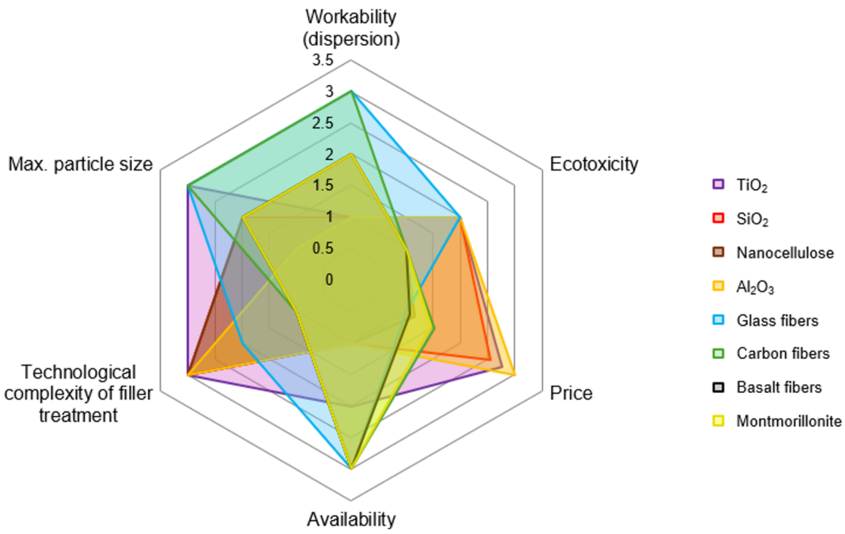

FIGURE 3. Analysis of filler and fibre parameters for the optimisation of wood adhesives.

price values (p) were recalculated to fit a scale from 1 to 3 (3), where number 1 represents the lowest and number 3 the highest value.

$$p_i = 1 + \frac{p_i - MIN(p) \times (3-1)}{MAX(p) - MIN(p)} \qquad (3)$$

The decision matrix in Table 4 shows that the criterion of workability (dispersion) has the highest weight in the selection of the filler or fibre. The use of reinforcing fillers and fibres can also have a negative impact on the mechanical parameters of the bonded joints in the case of the formation of aggregates of these particles in case of insufficient dispersion [20]. Due to the economic requirements in the field of adhesives, the price of fillers or fibres is also a very important criterion. In line with current environmental trends, it is essential to monitor the maximum dimensions of fillers or fibres, together with their ecotoxicity. An overview of the calculated weight values for each criterion is given in Table 4.

Figure 2 is a graphical representation of the results of the optimisation process. According to the given criteria, depending on the input parameters, glass (E) and carbon (F) fibres have the optimal values of the considered properties. In the case of the selection of glass fibres, it is necessary to verify the chemical resistance and compatibility of the fibres with the selected adhesives. It is also necessary to consider the environmental aspects of the production of these fibres and also the price, which in the case of primary production of carbon fibres may cause an economic disadvantage in their use.

4. CONCLUSION

By means of fillers and fibres, it is possible to modify the properties of adhesives such as viscosity, open workability time, mitigate the penetration of the adhesive into the wood structure, improve the resistance of the adhesive to volumetric changes of the wood adherend, as well as to increase the resistance of bonded joints in the case of moisture and temperature stress [2, 21–25].

When the reinforcement material is selected, it is necessary to take into account not only the price and the technological difficulty of the possible treatment of the fillers or fibres, but also the ecological character of the particle or fibre. Emphasis should also be directed to the potential profit of the filler as a by-product of production [21, 26].

- Based on calculations carried out by the quantitative pairwise comparison method using the Saaty matrix, glass and carbon fibres can be considered as the most suitable type of filler, which reached almost 100 %. They are relatively affordable and have low ecotoxicity.

- Another variant is the group formed by nanocellulose, basalt fibres and montmorillonite particles, which have achieved an average of 56 % suitability as reinforcing material for adhesives.
- TiO_2, SiO_2 and Al_2O_3 nanoparticles are not suitable reinforcing materials for adhesives due to their high cost, more difficult processability and increased possibility of toxicity.

This paper provides a basis for experiments aimed at modifying structural adhesives with reinforcing fillers and fibres.

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