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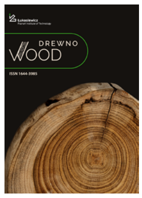

Modification of Casein Adhesives to Improve Moisture Resistance and Strength for Use in the Wood Industry

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This study investigated modifying casein adhesives to improve their moisture resistance and elasticity for use in the wood industry. Six different formulations were tested, including pure casein adhesive, polyvinyl acetate (PVAc) adhesive, and mixtures of the two, as well as modifications involving abietic acid, borax, and diethyl phthalate. While the modified casein adhesives met the minimum requirements according to DIN EN 205, they did not match the performance of the unmodified casein adhesive. Although the modified adhesives performed worse than the unmodified variants, they still demonstrate the potential for sustainable use of non-marketable milk as a source of raw materials.

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Introduction

As a protein derived from milk, casein offers a renewable and biodegradable alternative to synthetic adhesives, making it an attractive option for sustainable development. The production of irreversible casein adhesives using calcium hydroxide has been known for around 100 years. These adhesives were widely used in the wood industry, particularly for plywood and laminated wood, until the Second World War (Kollmann, 1962).

As early as the first half of the 20th century, studies were carried out to specifically improve the moisture resistance and strength properties of casein-based adhesives by adding suitable inorganic compounds, such as potassium water glass or copper chloride (Buttermann and Cooperrider, 1923). However, the adhesives and polymers developed at that time no longer meet current health, ecological, or performance requirements, particularly at high ambient moisture levels. Schwarzenbrunner *et al.* (2020) describe a method of increasing the moisture resistance of casein glues by using calcium carbonate and sodium silicate to achieve a gluing strength similar to that of samples glued with polyvinyl acetate. However, there is no information on long-term durability. Recent studies, such as that by Kowaluk and Wronka (2020), have explored the potential of biopolymer-based adhesives. These studies have highlighted the effectiveness of using biogenic raw materials to achieve comparable adhesive strengths to those of conventional products. This approach is in line with sustainable practices in the wood industry.

In addition to its use as an adhesive for bottle labelling, casein is now also used in food packaging (Audic et al., 2003). The challenge here is to counteract the brittleness of the biological plastic by adding suitable plasticisers, such as glycerol or sorbitol.

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Alongside modifying casein adhesives, many studies have aimed to improve the moisture resistance of adhesives derived from plant proteins (Heinrich, 2019). For instance, Huang and Sun (2000) demonstrated that adding sodium lauryl sulphate and sodium dodecylbenzene sulphonate significantly increased the shear strength and moisture resistance of soy protein adhesives.

This study aimed to demonstrate the use of biogenic agricultural residues (non-marketable milk) in sustainable wood adhesives. The effect of certain adhesive modifications on strength is to be demonstrated. These raw materials are readily available, given that milk production is the most important sector of the German agricultural industry. In 2022, Germany produced 32.4 million tonnes of cow's milk, accounting for over a fifth of the milk produced in the EU. According to the "Bioeconomy in Germany" report published by the Federal Ministry of Education and Research and the Federal Ministry of Food and Agriculture (BMBF and BMEL, 2014), around 1.9 million tonnes of non-marketable raw milk, which is currently disposed of or incinerated, is produced in Germany every year. Caseins account for around 2.7% of the proteins found in cow's milk. They can be extracted from milk for further processing.

This study aims to determine how additives can be incorporated into adhesives to impart additional functional properties. Building on the use of renewable raw materials in adhesives, the study aims to improve the properties of casein-based adhesives and plastics through suitable chemical and/or physical modifications. This will expand their potential applications in the wood industry and increase their market share.

Materials and methods

This study investigated five different casein-based adhesives in a tensile shear test according to DIN EN 302-1. To this end, six test specimens made of beech wood (*Fagus sylvatica* L.) with a moisture content of 8% and a thin adhesive joint were produced for each adhesive system. The specimens were prepared with dimensions specified by the DIN EN 302-1 standard, ensuring consistency and comparability in testing. Bonding was carried out on the tangential surface using a brush application at a pressing pressure of approximately 2 MPa. These tensile shear tests are evaluated in accordance with the DIN EN 205 standard. This standard was selected to evaluate the mechanical strength of the modified casein adhesives because that was the focus of the study. Although DIN EN 204 provides a classification based on moisture resistance, that was not the primary focus of this investigation.

The reference systems used were a conventional polyvinyl acetate (PVAc) adhesive and an irreversible casein adhesive (a waterproof adhesive) with hydrated lime and water glass (according to the approach of Buttermann and Cooperrider, 1923). Additionally, a 1:1 mixture of these two adhesives was investigated. Furthermore, three modifications of casein adhesive were tested in the tensile shear test: one with abietic acid (5 wt.%), one with borax (5 wt.%), and one with diethyl phthalate (7.5 wt.%). These additives were mixed into the casein adhesive immediately after production.

The addition of abietic acid (5 wt.%) was intended to increase resistance to biodegradation; borax (5 wt.%) was intended to act as a fire retardant; and diethyl phthalate (7.5 wt.%) was intended to act as a plasticiser to increase elasticity in the longitudinal tensile shear test. This study aims to investigate the effect of these modifications on the adhesive's strength properties.

The concentrations of these additives were chosen so that the effect of each modification would be clearly visible. This study is an initial exploration that aims to identify the potential impact of these additives on adhesive performance. Although diethyl phthalate is not a sustainable chemical, it was used to evaluate the overall impact of a plasticizer on adhesive properties. Future studies will focus on identifying more sustainable alternatives. Due to the limited sample size, the current study provides preliminary insights. Further research with larger sample sizes is planned to validate these findings more robustly.

Results and discussion

The results (Table 1) show that the tensile shear strength of the polyvinyl acetate adhesive, pure casein adhesive and the 1:1 mixture of the two components are comparable, ranging from 15.6 to 18.9 N/mm².

In accordance with DIN EN 205 (Adhesives – Wood adhesives for non-structural applications – Determination of bond strength of longitudinal bonds in the tensile test; German version EN 205:2016), classification in stress group D1 (indoor areas in which the wood moisture content does not exceed 15%) requires an average bond strength of at least 10 N/mm² after storage in a normal climate for 7 days. As shown in Table 1, all modified adhesives meet the minimum requirement, but their performance is notably lower than that of the unmodified casein adhesive. While all the modifications achieved these results on average, they are still below the measured values of the unmodified casein variant. As the casein adhesive is modified, the proportion of wood tear decreases. This indicates that failure primarily occurred in the

Table 1. 1 Mean values of the shear strengths [N/mm²] ($x_{\text{shear strength}}$) with the respective standard deviations ($s_{\text{shear strength}}$) and specification of the averaged percentage wood tear [%] ($x_{\text{wood tear}}$)

Adhesive	PVAc 100%	Casein 100%	Casein 50%; PVAc 50%	Casein 95%; Borax 5%	Casein 92.5%; diethyl phthalate 7.5%	Casein 95%; abietic acid 5%
$x_{\text{shear strength}}$	18.9	16.2	15.6	12.8	10.1	9.8
$s_{\text{shear strength}}$	1.6	1.0	1.4	1.4	0.3	1.9
$x_{\text{wood tear}}$	55	47	40	10	2	10

adhesive layer. The addition of diethyl phthalate was intended to enhance elasticity; however, this modification resulted in a decrease in tensile shear strength. This highlights the inherent trade-off between improving specific functional properties and maintaining mechanical performance. Future research should focus on optimizing additive concentrations to balance these aspects. One potential approach is to explore alternative plasticizers that offer improved sustainability without compromising adhesive strength.

It should be emphasised that the amount of additives in relation to the pure casein was very high (abietic acid: 28% by weight; borax: 28% by weight; diethyl phthalate: 40% by weight).

Conclusions

Preliminary work suggests that exploring the modification and combination possibilities of casein could strengthen this adhesive for use in the wood industry. The additives used here provide an initial indication of the quantities required and their effect on strength. The selection of modifiers was based on their capacity to enhance particular properties, such as elasticity and moisture resistance. However, further work is required to reduce the quantity of additives used here, which were added to the binder to fulfil certain functions, and to combine them with other additives. Thus, using casein as a renewable raw material could contribute to utilising biogenic residues and developing sustainable adhesives for the wood industry.

Conflict of interest

The author(s) declare(s) that there is no conflict of interest concerning the publication of this article.

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