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Protein-Based Adhesives for Particleboards

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Abstract

The main objective of this study was to elucidate the possibilities to use wheat gluten (WG) as a binder for particleboards, as well as soy protein isolate (SPI). The focus was on the effect of the adhesive formulation and the processing conditions, while the press parameters were kept constant. Some aspects of the dispersion and the preparation of the dispersions that were investigated are: the time (1, 3 or 5h) to prepare the dispersion, the temperature (room temperature, 50 or 80°C) during the preparation of the dispersions and the effect of storing (1, 2.5 or 4 days) the dispersions. Furthermore, the utilization of green particles versus dried particles was examined. The concentration (12, 16, 20 or 24%) of WG dispersion and the process for applying it to the particles was studied. Two application methods were evaluated, in application method 1 (appl. 1) all the dispersion was added to the particles in one step before the particles were dried. When application method 2 (appl. 2) was employed the dispersion was added in two steps, some of the dispersion was added before the drying and some after the drying. The considered board properties were internal bond (IB), thickness swelling (TS) and water absorption (ABS). The results show that in the case of SPI dispersion the temperature seems to not be of significant importance and it appears as if longer time to prepare the dispersion is beneficial. On the other hand for WG dispersion it looks as if a lower temperature is preferable for the preparation of the dispersion and that the time to prepare the dispersion is of no importance. Furthermore, storing the dispersions for more than one day before it was used as an adhesive for particleboards (PB) resulted in poorer boards. According to this study it is beneficial to use dried particles instead of green particles. Additionally, the results show that the interaction between the concentration of WG dispersion and how it is applied is a significant factor, considering the IB value. However, in general the two step process (appl. 2) is preferable.

Sammanfattning

Huvudsyftet med den här studien var att undersöka möjligheterna att använda vetegluten (WG) som bindemedel för spånskivor, även sojaproteinisolat (SPI) har undersökts. Fokus låg på effekten av formuleringen av bindemedlet och processbetingelserna, medan pressparametrarna hålls konstanta. En del parametrar som studerades med avseende på tillverkning av dispersionerna var: tiden (1, 3 eller 5 h) att bereda dem, vid vilken temperatur (rumstemperatur, 50 eller 80°C) de preparerades och effekten av att lagra (1, 2,5 eller 4 dagar) dispersionerna. Vidare, undersöktes användning av råspån kontra torkat spån. Koncentrationen (12, 16, 20 eller 24 %) för WG-dispersionerna och processen för belimning med dessa dispersioner utvärderades. Två applikationsmetoder testades, i applikationsmetod 1 tillsattes hela mängden dispersion i ett steg till spånet innan det torkades. I applikationsmetod 2 tillsattes dispersionen i två steg en del tillsattes innan torkning och resten tillsattes efter torkning. Skivegenskaperna som utvärderades var, tvärdrag (IB), svällning (TS) och absorption (ABS). Resultaten visade att det kan vara fördelaktigt att tillreda SPI-dispersioner under en längre tid och det verkar inte som att temperaturen är en viktig parameter. Däremot förefaller det som att lägre temperatur är gynnsamt när en WG-dispersion ska blandas och att tiden inte är av signifikant betydelse. Vidare, förefaller det som att lagring längre än en dag av dispersionerna har en negativ effekt på deras förmåga som bindemedel för spånskivor. Enligt den här studien är det fördelaktigt med avseende på skivegenskaper att använda torkat spån istället för råspån. Resultaten visar att samverkan mellan koncentration och applikationsmetod kan vara en signifikant faktor, med avseende på IB-värdena. Dock verkar det överlag som att tvåstegsbelimningen är fördelaktig.

List of papers

- I. **Protein-based adhesives for particleboards**
S. Khosravi, F. Khabbaz, P. Nordqvist and M. Johansson, *Industrial Crops and Product*, 32 (2010) 275-283.
- II. **Protein-based adhesives for particleboards – effect of application process**
S. Khosravi, P. Nordqvist, F. Khabbaz, and M. Johansson, submitted to *Industrial Crops and Product* (2011).

The contributions of the author of this thesis to the appended papers are:

- I. Most of the experimental work and the preparation of the manuscript.
- II. Most of the experimental work and the preparation of the manuscript.

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1. Aim of study

The most commonly used adhesives for particleboard production are formaldehyde-based amino resins. Although technically working well, there are two main issues with these resins, they are petroleum based and they have to be tailor made in order to obtain sufficiently low formaldehyde emission from the produced boards. Therefore, it is valuable to have adhesives based on renewable resources that are formaldehyde free. However, the same technical performance has to be obtained. One attractive option for this application is plant proteins such as, protein from wheat and soy bean [1].

The overall objective of this thesis is to evaluate the possibilities to employ protein-based adhesives for particleboard production. The study aims to reveal details on which structural parameters and processing conditions that affect the adhesive performance in particleboards. In this study wheat gluten (WG) and soy protein isolate (SPI) were evaluated. More specifically the aim of this study was to investigate the impact of the adhesive formulation and the gluing process, for these protein-based adhesives.

2. Introduction

Proteins have been used as adhesives for thousands of years. Protein adhesives for more recent wood applications were commonly made with soybean in combination with blood and casein [2]. These adhesives were mostly employed for interior plywood, which did not require high moisture resistance. Once protein-based adhesives were the dominant wood adhesives however, since the 1960's they have for the most part been replaced by petroleum-based adhesives. Most petroleum-based adhesives are superior in strength and water resistance, furthermore, they are also more cost efficient. Nevertheless, due to increased environmental awareness and increased oil prices once again protein-based adhesives are of interest.

2.1 Particleboards

Particleboard is a wood-based composite that is used for many applications such as, for furniture, in flooring and as panels. Particleboards consist of wood particles that are glued together at high temperature and high pressure. The wood particles can consist of sawdust, saw shavings, recycled wood, agricultural residue etc. The particles are separated based on size, after they have been dried. The sizes of the particles are of great importance and will influence the properties of the final product. Normally, particleboards have three layers, a core layer with coarser particles and a lower density and two surface layers with finer particles and higher densities [3-4].

Most commonly formaldehyde amino based adhesives are employed for the production of particleboards. The type of adhesive used depends on the requirements of the boards, for example, to produce water resistant boards, normally MUF (melamine-urea-formaldehyde) resins are used, and for non water resistant boards UF (urea-formaldehyde) or UMF (urea-melamine-formaldehyde) resins are used. Today UMF resins are employed more frequently in order for the boards to have sufficiently low formaldehyde emission [5].

The adhesive and other additives, such as hardener and wax, are added, through nozzles, to the particles in the gluing machines. In order to obtain good board properties it is very important to have a good distribution of the adhesive on the particles. One of the adhesive parameters that influence the distribution is the viscosity of the adhesive. After the gluing, the particles arrive to the forming station; see Figure 1 for an outline of a particleboard plant. The ratio between the surface layers and the core layer, and the amount of fine material in the core layer can be regulated in the forming station. Usually, the formed mat goes to the pre-press before entering the hot press where the mat is subjected to high temperature and high pressure, in order to produce boards. The press time and temperature are very important, since it is necessary to get a high enough temperature in the core to cure the adhesive, at the same time as the temperature at the surface should not be too high to avoid thermal degradation. After the press, the boards go to a cooling wheel, and finally they are sanded and cut [3-4].

Figure 1 – Outline of a particleboard plant [6].

There are several tests used to determine the properties of particleboards. Some of the most common properties normally determined are: density of the board (dens.), internal bond (IB), modulus of rupture (MOR), modulus of elasticity (MOE), thickness swelling (TS), water absorption (ABS), surface soundness (SS), perforator values (PV), and the emission of formaldehyde from the boards.

The requirements on particleboard properties depend on which end-use application they are intended for. There are standards that define the requirements where one typical standard for furniture for example is, EN 312 quality P2, which states that the properties for a 16 mm board should be as follows: IB 0.35 MPa, MOR 13 MPa, MOE 1500 MPa and SS 0.8 MPa.

2.2 Proteins

Proteins are composed of amino acids, the amino (-NH_2) - and carboxyl (-COOH) groups form peptide bonds and tie the amino acids together. There are about 20 different amino acids that can be combined to form a protein. The structure and the properties of the protein depend on the protein source. Proteins are divided into two major categories, fibrous proteins and globular proteins. Fibrous proteins are held together as fibrils by hydrogen bonds and they are insoluble in water. Globular proteins are folded and they are more compact than fibrous proteins. Principally, a combination of intramolecular secondary bonding forces determines the shape of the protein [7].

To describe and understand the structure of proteins it is necessary to study, at least four levels, the primary, the secondary, the tertiary and the quaternary level. The primary level describes the amino acid sequence. The regularity of the repeating conformations of the protein relates to the secondary level; examples of secondary structures are α -helix and β -sheet. The tertiary level describes the three-dimensional structure of a protein molecule. Aggregates of the protein with other proteins or other molecules relates to the quaternary level [8-9].

2.3 Wheat gluten

Wheat protein can be divided into four groups, albumins, globulins, gliadins and glutenins. Albumins and globulins are water and salt soluble, while gliadins are soluble in alcohol and glutenins are dispersible in dilute acids or bases. Gliadins and glutenins correspond to approximately 85% of the total protein content in wheat [10].

Wheat gluten (WG) is a byproduct from the production of wheat starch. During this process the wheat is washed, and albumin and globulins that are soluble in water and salts are washed out. Wheat gluten consists of almost equal amounts of glutenins and gliadins and these consist of large amounts of proline and glutamine. Wheat gluten is a hydrophobic protein due to the high content of non-polar amino acids. Wheat gluten consists of ca. 80% proteins and some polysaccharides, lipids and minerals. The isoelectric point for wheat gluten is around pH 7.3 [10-14].

The gliadin fraction in wheat gluten is soluble in alcohol and the molecular weight is 30 000-74 000 g/mol. Furthermore, the gliadins constitute the viscous components in wheat gluten, while the glutenin fraction contributes to the elastic properties. Glutenins are dispersible in dilute acids and alkaline solutions and have a molecular weight higher than 10^7 g/mol [10, 12].

Today this visco-elastic protein is mainly used in the bakery industry but it is also being used as, for example, films for food packing applications [11, 14-15].

2.4 Soy protein

Soy proteins are mainly globular and have a hydrophilic character. The isoelectric point (pI) is at approximately pH 4.5. Soy protein can be divided into two groups, globulins which is the major fraction, and albumins. Globulins are soluble in salt solutions, while albumins are soluble in water. Globulins can be further categorized into glycinin and conglycinin. Glycinins have a molecular weight between 200 000-400 000 g/mol and consist of six acid-base subunits that are joined by disulfide bonds. Conglycinins have a molecular weight between 100 000-200 000 g/mol and consist of three subunits that are coupled by hydrogen bonds. Conglycinins are less hydrophobic than the glycinins [1, 16-17].

Soy protein isolate has a high concentration of protein, approximately 92%, and is therefore more expensive than for example soy flour (48% protein). The higher the protein content the higher is the price since more processing is required [17].

3. Experimental

Bellow follows the main information about the performed experiments. For complete details see papers I and II.

3.1 Material and equipment

Wheat gluten (WG) Reppe Vital from Lantmännen Reppe AB was used in this study. The protein content is ca 85% and the particle size is equal or larger than 50 μm for 83% of the sample, according to the supplier.

The employed soy protein isolate (SPI) Soy Pro 900 was supplied from Roquette (Qingdo Crown Imp. & Exp. Corp. Ltd). According to the supplier the protein content is approximately 90% and at least 96% of the sample has a particle size smaller than 150 μm .

The wood particles were kindly supplied by ACB Laminat AB and Byggelit AB. Two types of particles were used dried core-particles and green particles. The particles consist mainly of spruce, some pine and a small part of other wood materials. Particle size distribution are reported in papers I and II.

An upright drilling machine was utilized for the gluing of the particles, see Figure 2. It consists of a container and an agitator. The container diameter is 25.5 cm and its height is 35 cm. Furthermore, there is a baffle inside the container in order to improve the mixing.

Figure 2 – The upright drilling machine employed to glue the particles.

The dryer is a large container where the particles are tumble-dried with the help of an air stream that passes through the middle of the container. The outlet temperature of the air is 40-50°C.

An IR-scale (Mettler LP 16, PM460) was used for determining the moisture content of the glued particles.

A hydraulic press, Ake (Mariannelund Sweden), with two pistons was used as a hot press (Type HPB-750X600X1X71). The piston area is 904 cm² (the area of each piston is 452 cm²) and the press table dimension is 76 cm x 61 cm.

The forming station consist of two parts, a wagon on which the mat is formed and a feeder. The particles are brushed down from the feeder to a plate on the wagon, which moves back and forth. The forming station is built in house.

3.2 Preparation and analysis of the dispersions

For the preparation of the wheat gluten (WG) dispersions sodium hydroxide (0.1 M, pH 13) was used as dispersing agent. The WG was added portion-wise to a beaker containing the sodium hydroxide, a mechanical agitator was employed. In the case of the SPI dispersions the SPI was dispersed in water otherwise the same procedure was used.

An oil bath was utilized when dispersions were prepared at higher temperature than room temperature. Furthermore, the dispersions were used the day after they had been prepared, if not otherwise stated. For some of the dispersions the solids content were determined gravimetrically.

3.3 Gluing of the wood particles and the particleboard pressing

The particles were glued in the upright drilling machine and the blending time was 2.5 minutes. If the theoretical moisture content of the glued particles were above 16% they were dried before a mat was formed and pressed. In some cases dispersion or water was added to the glued particles after they had been dried and before the mat was formed. The added amount of dry WG or SPI was 10% per dry particles in all the experiments.

The dimensions of the produced particleboards were 30 cm x 30 cm x 1.6 cm. The press temperature was 185°C and the press time was 2.7 minutes. The pressure cycle was: 150 kg/cm² for 0.5 min, 40 kg/cm² for 1.7 min and 2 kg/cm² for 0.5 min. A pressure of 2 kg/cm² is required to keep the press closed.

3.4 Board evaluation

3.4.1 Sample preparation of the boards

The boards were conditioned for one week at 20 °C and a relative humidity (RH) of 65 %. In order to be able to evaluate the results statistically six pieces (5 cm x 5 cm), of each sample were analyzed for each property.

3.4.2 Evaluated board properties

The density (dens.), internal bond (IB), thickness swelling (TS), and water absorption (ABS) properties were analyzed. The IB and the TS were measured according to the European standards SS-EN 319 and SS-EN 317. For determining the ABS, the same pieces that were used to measure TS were weighted, and the increase in percent is defined as the ABS of the board. Higher IB values are desirable, while in the case of TS and ABS lower values are preferable.

3.4.3 Evaluation with software SIMCA-P+ from Umetrics AB

For multivariate data analysis of the experiments conducted according to a factorial design, the software SIMCA-P+ from Umetrics AB (Umeå, Sweden), was used.

3.5 Test series

In this study several aspects of employing protein-based adhesives for producing particleboards (PB) were investigated. More specifically, the type of particles used, the formulation of the adhesives and for WG dispersions the gluing process, were explored.

3.5.1 Binder system –effect of dry protein versus water dispersions

In this set of experiments the proteins were used as dry protein powder (DP) and as protein dispersions. All of these dispersions were prepared at room temperature (RT) during 2h. The solids content of the SPI dispersion (SPIdisp-RT-2h) was 12% and it was 20% for the WG dispersion (WGdisp-RT-2h). The dry protein powder was employed in combination with different amounts of water or in some cases together with the protein dispersion. For more details regarding the gluing and the drying of the particles see Table 1.

Table 1 – The gluing conditions of the particles, for each experiment the addition of WG or SPI was 10% /dry particles.

Sample	Water	Dispersion		Dry protein		Drying time (min)	Moisture content glued particles (%)
	(g)	type	amount (g)	type	amount (g)		
Water + SPI	110	-	-	SPI	85	-	13.0
Water + WG	110	-	-	WG	85	-	13.9
SPIdisp-RT-2h + SPI	-	SPIdisp-RT-2h	125	SPI	70	-	9.9
WGdisp-RT-2h + WG	-	WGdisp-RT-2h	137	WG	57	-	11.1
SPIdisp-RT-2h + dry	-	SPIdisp-RT-2h	706	-	-	15	15.0
WGdisp-RT-2h + dry	-	WGdisp-RT-2h	423	-	-	25	11.4
Water + SPI + dry	622	-	-	SPI	85	10	11.2
Water + WG + dry	340	-	-	WG	85	19	13.2

A board was also made with UF 1115 (65%), which is a commercial resin from Casco Adhesives AB; 6.4% dry resin was added per dry particles and ammonium chloride (20%) was utilized as hardener, 4% dry hardener was added per dry resin.

3.5.2 Binder system, time and temperature for the preparation of the dispersions

The following experiments were conducted according to a factorial screening design, therefore, the results were analyzed with SIMCA-P+. The effect of the temperature (RT, 50 or 80°C) during the preparation of the dispersion and the time (1, 3 or 5h) to prepare the dispersion was investigated. Table 2 presents specific information on how the dispersions were prepared and their solids content. Furthermore, experiments were conducted with protein dispersions as the adhesive and protein dispersion in combination with dry protein powder as the adhesive. Table 3 provides details regarding the gluing and the drying of the particles.

A second set of experiments were made on a second occasion, with the same batch of particles, however, one should keep in mind that during this time the particles have aged. The dispersions were prepared at room temperature, 50°C or 80°C, during 3h. Two boards were made with each dispersion.

Table 2 – The conditions during which the dispersions were prepared.

Dispersion	Protein	Temperature (°C)	Stirring time (h)	Solids content (%)
SPIdisp-RT-1h	SPI	RT	1	11.9
WGdisp-RT-1h	WG	RT	1	20.1
SPIdisp-RT-5h	SPI	RT	5	11.8
WGdisp-RT-5h	WG	RT	5	19.5
SPIdisp-50°C-3h	SPI	50	3	12.7
WGdisp-50°C-3h	WG	50	3	20.5
SPIdisp-80°C-1h	SPI	80	1	12.4
WGdisp-80°C-1h	WG	80	1	20.0
SPIdisp-80°C-5h	SPI	80	5	15.1
WGdisp-80°C-5h	WG	80	5	25.7
SPIdisp-RT-3h	SPI	RT	3	12.0
WGdisp-RT-3h	WG	RT	3	20.0
SPIdisp-50°C-3h (2)	SPI	50	3	12.1
WGdisp-50°C-3h(2)	WG	50	3	20.2
SPIdisp-80°C-3h	SPI	80	3	12.6
WGdisp-80°C-3h	WG	80	3	21.9

See Table 3 for further information about the gluing and the drying of the particles. This second set of experiments, were analyzed together with the experiments conducted on the first occasion in SIMCA-P+. Moreover, it was presumed that room temperature equals 20°C.

Table 3 – The gluing conditions of the particles, for each experiment the addition of WG or SPI was 10% /dry particles.

Sample	Water	Dispersion	amount (g)	Dry protein		Drying time (min)	Moisture content glued particles (%)
	(g)	type		type	amount (g)		
SPIdisp-RT-1h + dry	-	SPIdisp-RT-1h	706	-	-	14	12.9
SPIdisp-RT-1h + SPI	-	SPIdisp-RT-1h	125	SPI	70	-	11.1
WGdisp-RT-1h + dry	-	WGdisp-RT-1h	423	-	-	7	13.9
Water + WGdisp-RT-1h + WG	50	WGdisp-RT-1h	75	WG	70	-	12.0
SPIdisp-RT-5h + dry	-	SPIdisp-RT-5h	718	-	-	13	12.0
SPIdisp-RT-5h + SPI	-	SPIdisp-RT-5h	125	SPI	70	-	10.2
WGdisp-RT-5h + dry	-	WGdisp-RT-5h	434	-	-	7	13.0
Water + WGdisp-RT-5h + WG	48	WGdisp-RT-5h	77	WG	70	-	10.9
SPIdisp-50°C-3h + dry (1)*	-	SPIdisp-50°C-3h	706	-	-	11	17.0
SPIdisp-50°C-3h + dry (2)*	-	SPIdisp-50°C-3h	706	-	-	13.5	11.0
SPIdisp-50°C-3h + dry (3)*	-	SPIdisp-50°C-3h	706	-	-	13	15.4
WGdisp-50°C-3h + dry (1)*	-	WGdisp-50°C-3h	413	-	-	5	12.9
SPIdisp-80°C-1h + dry	-	SPIdisp-80°C-1h	683	-	-	12	15.0
Water + SPIdisp-80°C-1h + SPI	4	SPIdisp-80°C-1h	121	SPI	70	-	12.2
WGdisp-80°C-1h + dry	-	WGdisp-80°C-1h	423	-	-	4.5	13.0
Water + WGdisp-80°C-1h + WG	50	WGdisp-80°C-1h	75	WG	70	-	10.0
SPIdisp-80°C-5h + dry	-	SPIdisp-80°C-5h	561	-	-	8	14.1
Water + SPIdisp-80°C-5h + SPI	26	SPIdisp-80°C-5h	99	SPI	70	-	11.0
WGdisp-80°C-5h + dry	-	WGdisp-80°C-5h	329	-	-	3	14.0
Water + WGdisp-80°C-5h + WG	67	WGdisp-80°C-5h	58	WG	70	-	12.0
SPIdisp-RT-3h + dry (1)*	-	SPIdisp-RT-3h	706	-	-	14.5	13.0
SPIdisp-RT-3h + dry (2)*	-	SPIdisp-RT-3h	706	-	-	20**	16.0
WGdisp-RT-3h + dry (1)*	-	WGdisp-RT-3h	423	-	-	6	15.4
WGdisp-RT-3h + dry (2)*	-	WGdisp-RT-3h	423	-	-	6.5	13.7
SPIdisp-50°C-3h + dry (4)*	-	SPIdisp-50°C-3h (2)	700	-	-	13	15.8
SPIdisp-50°C-3h + dry (5)*	-	SPIdisp-50°C-3h (2)	700	-	-	13	14.6
WGdisp-50°C-3h + dry (2)*	-	WGdisp-50°C-3h(2)	419	-	-	6	13.3
WGdisp-50°C-3h + dry (3)*	-	WGdisp-50°C-3h(2)	419	-	-	6	13.5
SPIdisp-80°C-3h + dry (1)*	-	SPIdisp-80°C-3h	672	-	-	12	12.6
SPIdisp-80°C-3h + dry (2)*	-	SPIdisp-80°C-3h	672	-	-	11.5	13.4
WGdisp-80°C-3h + dry (1)*	-	WGdisp-80°C-3h	387	-	-	6	12.2
WGdisp-80°C-3h + dry (2)*	-	WGdisp-80°C-3h	387	-	-	5.5	10.9

* When several boards were produced with the same recipe, the numbers in the brackets distinguishes them from each other.

** The suction out of the dryer was turned off sometime during the first 14 minutes, therefore, the glued chips were dried for 6 additional minutes.

3.5.3 Type of particles and storage of the dispersions

In this set of experiments the influence of the type of particles, green particles (gp) or dried particles (dp) were looked into. The green particles were divided into two parts, and half of it was dried. Furthermore, the effect of storing the dispersions, 1, 2.5 or 5 days, was evaluated. The dispersions were prepared at room temperature and during 1h. SIMCA-P+ was used to evaluate the results. Table 4 provides the details for these experiments.

Table 4 – The gluing conditions of the particles, for each experiment the addition of WG or SPI was 10% /dry particles. The moisture content, of the glued particles, was determined after they had been dried.

Sample	Particles		Dispersion		Moisture content glued particles (%)
	type	amount (g)	type	amount (g)	
gp + SPIdisp-1d + dry	green	1695	SPIdisp-RT-1h	703	8.9
gp + WGdisp-1d + dry	green	1695	WGdisp-RT-1h	422	13.4
dp + SPIdisp-1d + dry	dried	864	SPIdisp-RT-1h	703	11.9
dp + WGdisp-1d + dry	dried	864	WGdisp-RT-1h	422	14.0
dp + SPIdisp-2.5d + dry (1)*	dried	864	SPIdisp-RT-1h	703	12.9
dp + SPIdisp-2.5d + dry (2)*	dried	864	SPIdisp-RT-1h	703	11.6
dp + SPIdisp-2.5d + dry (3)*	dried	864	SPIdisp-RT-1h	703	10.7
gp + SPIdisp-4d + dry	green	1695	SPIdisp-RT-1h	703	14.4
gp + WGdisp-4d + dry	green	1695	WGdisp-RT-1h	422	8.2
dp + SPIdisp-4d + dry	dried	864	SPIdisp-RT-1h	703	12.1
dp + WGdisp-4d + dry	dried	864	WGdisp-RT-1h	422	15.6

* When several boards were produced with the same recipe, the numbers in the brackets distinguishes them from each other.

3.5.4 Effect of the gluing process - application method 1 and 2

In this test series the focus was on wheat gluten dispersions and how the concentration of the dispersions and the application method influences the board properties. Dispersions with different concentrations were used and two application methods were employed for the gluing of the particles. When application method 1 (appl. 1) was used all the dispersion was added to the particles and then the particles were dried before a mat was formed with the help of the forming station and the board was pressed. Application method 2 is a two step process. In the first step 80% of the total amount of the dispersion is added to the particles and in the second step, after the particles have been dried, the rest of the dispersion is added before a mat is formed and pressed. See Table 5 for further information regarding the execution of the experiments.

A particleboard was made with a commercial UMF resin (UMF 0872 (68%)) from Casco Adhesives AB. The resin dosage was 6.4% dry resin per dry particles and 4% of dry hardener (ammonium chloride) per resin was added.

Table 5 – The gluing of the particles, for each experiment the addition of WG was 10% /dry particles. The moisture content, of the glued particles, was determined before the mat was formed and pressed.

Sample	Dispersion conc. (%)	Step 1 amount (g)	Step 2 amount (g)	Moisture content glued chips (%)
WG-12_1 (1)	12	811	-	13.0
WG-16_1 (1)	16	608	-	13.3
WG-20_1 (1)	20	487	-	12.9
WG-24_1 (1)	24	405	-	11.5
WG-12_2 (1)	12	649	162	14.0
WG-16_2 (1)	16	487	122	13.2
WG-20_1 (2)	20	487	-	11.5
WG-20_2 (1)	20	389	97	14.1
WG-20_2 (2)	20	389	97	12.2
WG-20_1 (3)	20	487	-	11.2
WG-24_2 (1)	24	324	81	12.7
WG-20_2 (3)	20	389	97	15.3
WG-24_2 (2)	24	324	81	n.d
WG-12_1 (2)	12	811	-	n.d
WG-20_2 (4)	20	389	97	n.d
WG-16_1 (2)	16	608	-	n.d
WG-16_2 (2)	16	487	122	n.d
WG-20_1 (4)	20	487	-	n.d
WG-12_2 (2)	12	649	162	n.d
WG-24_1 (2)	24	405	-	n.d

The first number in the sample name points to the concentration (12, 16, 20 or 24 %) of the dispersion and the second number identifies the application method (method 1 or 2). When several boards were made with the same recipe, the numbers in the brackets distinguishes them from each other. n.d stands for no data.

3.5.4.1 Effect of water

Two control experiments were conducted with WG 12%, in order to study the effect of water in the two step process. One board was made according to application method 2, while the other board was made by adding all the dispersion in one step before the drying of the particles and then water was added in a second step. The amount of water corresponds to the amount of water that would be added in the second step of the two step process via the dispersion, see Table 6.

Table 6 – The gluing of the particles, for each experiment the addition of WG was 10% /dry particles. The moisture content, of the glued particles, was determined before the mat was formed and pressed.

Sample	Dispersion conc. (%)	Step 1 amount (g)	Step 2 amount (g)	Water (g)	Moisture content glued chips (%)
WG-12_2 (3)	12	649	162	-	n.d
WG-12_W	12	811	-	142	13.0

The first number in the sample name indicates the concentration (12 %) of the dispersion and then the application method is specified, 2 stands for application method 2 and W stands for addition of water in a second step. When several boards were made with the same recipe, the numbers in the brackets distinguishes them from each other. n.d stands for no data.

4. Results and discussion

4.1 Binder system – effect of dry protein versus water dispersions

The experiments described in 3.5.1 were conducted as a starting point to find out which factors are important, in order to have relevant information for designing the following experiments. These experiments were made using dry protein powder (DP) in combination with water, protein dispersion, or protein dispersion together with dry protein powder.

When a dispersion or in some cases dry protein powder in combination with water were employed as an adhesive it was necessary to dry the particles before pressing the boards. When dry protein powder combined with a dispersion or with small amounts of water were used, it is not necessary to dry the particles, which is an advantage from a process point of view. The results are presented in Table 7.

Table 7 – Summary of the results from the experiments described in section 3.5.1. Higher IB values are sought, in contrast to TS and ABS where lower values are preferable. The values are means $\pm$ standard deviation.

	Dens. (kg/m ³)	IB (MPa)	ABS (%)	TS (%)	MC (%)
Water + SPI	620 $\pm$ 51	0.08 $\pm$ 0.05	178 $\pm$ 16	82 $\pm$ 4	13.0
Water + WG	605 $\pm$ 39	0.06 $\pm$ 0.03	210 $\pm$ 10	100 $\pm$ 7	13.9
SPIdisp + SPI	662 $\pm$ 14	0.18 $\pm$ 0.02	186 $\pm$ 20	90 $\pm$ 3	9.9
WGdisp + WG	569 $\pm$ 37	0.19 $\pm$ 0.04	187 $\pm$ 2	83 $\pm$ 9	11.1
SPIdisp + dry	602 $\pm$ 44	0.17 $\pm$ 0.05	158 $\pm$ 12	68 $\pm$ 3	15.0
WGdisp + dry	572 $\pm$ 29	0.35 $\pm$ 0.06	165 $\pm$ 15	63 $\pm$ 7	11.4
Water + SPI + dry	612 $\pm$ 63	-	-	-	11.2
Water + WG + dry	-	-	-	-	13.2
UF 1115	700 $\pm$ 48	0.82 $\pm$ 0.13	91 $\pm$ 3	37 $\pm$ 3	7.0

Using UF resin as the adhesive resulted in a board with better properties compared to the boards where protein-based adhesives were utilized. The exceptionally high internal bond (IB) value can to some extent be explained by the high density. Most likely, this board had a higher density due to the fact that the particles glued with UF resin had much higher tack. This means that the edges of the mat are kept together much better and less particles fall off the mat.

Even if the results with the protein-based adhesives are not as good as the results with the UF resin, these resins do show potentials. The results reveal that it is beneficial to disperse the proteins instead of using them as dry protein powder. Moreover, when a high amount of water was added to the particles before, the addition of dry protein powder, the board properties were very poor. In the case of WG the board delaminated and in the case of SPI it was

impossible to measure the board properties, due to very poor glue bonds. This indicates that to obtain the best gluing properties for these proteins, they need to be dispersed. It is not enough to just have water present together with the protein.

When SPI dispersion was used and the glued particles were dried (SPIdisp + dry), the internal bond is similar to when SPI dispersion in combination with SPI powder (SPIdisp + SPI) was employed. A suggested explanation is that in the first case the density of the board is lower, which can result in lower IB values [4]. Additionally, there is a difference in the moisture content of the glued particles.

4.2 Binder system, time and temperature for the preparation of the dispersions

To evaluate the influence of each factor on the board properties, for this set of experiments software SIMCA-P+ was used. The four controlled factors were: type of protein, SPI or WG; binder system, dispersion and drying or dispersion and dry protein powder (DP); temperature during the preparation of the dispersion, RT (20°C), 50°C or 80°C; time for the preparation of the dispersion, 1, 3 or 5 h. Additionally, two factors that could not be controlled entirely, although determined, were: the density of the boards and the moisture content of the glued particles before the hot press. The three responses were: IB, TS and ABS.

In a summary of fit plot R2Y describes the variation in Y (the responses) that can be correlated to X (the factors), and Q2 describes how large part of Y (the responses) that can be predicted by the model according to cross validation. The higher R2Y and Q2 the better model; furthermore, the difference between the R2Y and the Q2 value should not be too large. If the difference is too large it means that the model is over estimated. A summary of fit plot obtained by PLS (projection to latent structures by means of partial least square analysis), may include several components (Comp). Each component provides information that explains the variation in the data. The difference between R2Y and Q2 in component 1 and 2 shows the amount of explanation found in component 2.

The summary of fit plot in Figure 3 shows that a good model is obtained for the different responses, based on the observations. In view of this fact, this model can be used to evaluate how the different factors influence the properties of the particleboards.

Figure 3 – Summary of fit plot for a model based on all experiments in section 3.5.2.

A scatter plot illustrates the distribution within a set of experiments and reveals if there are group formations. In Figure 4 six groups can be seen based on the type of protein, binder system, and when the experiments were conducted. The boxes represent the experiments where a dispersion was used in combination with the protein powder. The samples where only a dispersion was employed as adhesive are indicated as triangles. Furthermore, the boards that were produced on the second occasion are denoted by the circles. Each one of these three groups can be divided into two sub-groups, depending on the type of protein used; the samples more to the left within each group represent WG.

Figure 4 – Scatter plot for all experiments in section 3.5.2, showing the formations of groups depending on formulation, type of protein and when the experiments were conducted. Dispersion in combination with protein powder (■), pure dispersions, first occasion (▲), pure dispersions, second occasion (○).

Figure 5 shows a coefficient plot which represents the different factors and reveals their impact on a response. Each bar represents different factors and the size of the bar illustrates the importance of the factor; the larger the bar, the more important is the factor. If a qualitative factor, such as type of protein, is positive it means that the response value will increase with the use of this factor. However, if it is negative the response value will decrease. In the cases of quantitative factors, such as the temperature and the time for the preparations of the dispersions, a positive factor means that the higher value of the factor the higher will the value of the response be. On the other hand if the factor is negative, the higher the absolute value of the factor the lower will the value of the response be. If the line representing the standard deviation crosses the zero value it means that this factor is not significantly important. The different components in summary of fit plots will have different coefficient plots; the coefficient plot for the first components will be presented, unless the other components provide more information.

The factor with the main influence on the internal bond (IB) value is the binder system, according to Figure 5. It is an advantage to use the proteins as dispersions. Furthermore, another significant factor is on which occasion the experiments were conducted; it seems as if the boards made on the second occasion had a higher IB values. A possible explanation to this might be the fact that during the time that has passed between the two occasions the particles have aged, which changes the properties of the particles. The results indicate that it is beneficial if the glued particles have higher moisture content; nevertheless, most likely this is only true to a certain level and higher moisture content will lead to delamination of the boards, due to steam blisters. Additionally, it seems like SPI may be better than WG considering the internal bond property; one plausible explanation can be that SPI (protein content ca. 90%) contains a higher amount of protein than WG (protein content ca. 85%).

Figure 5 – Coefficient plot (first component) for internal bond (IB), a high value for IB is desirable.

Also in the case of the thickness swelling of the boards, the principal factor is the binder system; it is favorable to use dispersions (see Figure 6). Likewise, higher moisture content has a positive effect on the TS and the experiments performed on the second occasion had lower TS values.

Figure 6 – Coefficient plot (first component) for thickness swelling (TS), a lower value for TS is preferable.

Figure 7 shows that the same factors are of significant importance for the ABS as for the TS. This means that it is preferable to use a dispersion, to have higher moisture content and to employ the particles that had been aged (second occasion).

Figure 7 – Coefficient plot (first component) for absorption (ABS), a lower value for ABS is preferable.

A clear difference in the results can be seen for the two occasions, for all the evaluated board properties. This shows that the aging of the particles will affect the board properties. Therefore, it is important to keep in mind that it is not always possible to compare experiments conducted with different batches of particles or even with the same batch of particles, due to the fact that the properties of the particles will differ.

There is a possibility that SPI and WG are affected in different ways by the varying factors during the preparation of the dispersions, therefore, they were also evaluated separately. Only the experiments from the first occasion were analyzed and the time for drying the particles was added as a factor. However, the binder system and the time for drying the particles are bound to each other. Nevertheless, including drying time as a factor, results in an improvement of the model, since it contributes to explaining the variations in the response further.

A good model was obtained for the experiments with SPI and it can describe the influence of the factors on the responses. The summary of fit plot (PLS) has two components and the cumulative R^2Y value is 0.89 and Q^2 value is 0.78. Figure 8 implies that longer time to prepare the SPI dispersion has a positive effect on the IB value. However, these experiments were conducted as screening experiments and do not reveal the optimal time for preparing the dispersions. On the other hand, the temperature for preparing the dispersion was not of significant importance in the investigated temperature range. Furthermore, both the binder system and the drying time are significant factors; these two factors are closely connected since the drying time depends on the used binder system. It is advantageous to employ the protein as a dispersion. For the thickness swelling and the absorption no new information was obtained.

Figure 8 – Coefficient plot (second component) for internal bond (IB), in the cases of SPI. Higher value for IB is desirable.

For the experiments with WG the TS response was removed in order to improve the model. The summary of fit plot (PLS) for these experiments has one component and the cumulative

R²Y value is 0.94 and Q² value is 0.91. Figures 9 and 10, reveals that the temperature influences the IB and ABS values; lower temperature seems to be advantageous for the evaluated temperature interval. In contrast the time to prepare the dispersions appears to be a trivial factor in the case of WG, in the investigated interval.

Figure 9 – Coefficient plot (first component) for internal bond (IB), in the cases of WG. Higher value for IB is preferable.

Figure 10 – Coefficient plot (first component) for absorption (ABS), in the cases of WG. Lower value for ABS is preferable.

4.3 Type of particles and storage of the dispersions

This set of experiment was, also evaluated with software SIMCA-P+. The three controlled factors were: Type of protein (SPI or WG), type of particles (dried particles or green particles), and storage of the dispersion (1, 2.5 or 4 days). Additionally, two factors that could not be controlled entirely, although determined, were: the density of the boards, and the moisture content of the glued particles after drying. The three responses were: IB, TS and ABS.

In three out of four cases when green particles had been used it was impossible to measure the TS and ABS, since the samples fell apart in water. Therefore, these three samples were removed from the model. To further improve the model the response ABS was excluded. The summary of fit plot (PLS) has four components and the cumulative R²Y value is 0.99 and Q² value is 0.94. This model can be used to explore how the factors affect the board properties.

The coefficient plots shows that the type of particles is not a significant factor, see Figures 11 and 13. Nevertheless, most experiments using green particles were excluded due to poor properties; consequently, it is reasonable to assume that the utilization of green particles is a disadvantage compared to using dried particles.

Figure 11 – Coefficient plot (first component) for internal bond (IB), a high value for IB is desirable.

In addition, it is suggested that longer storage time (days) of the protein dispersions has a negative effect on the internal bond values. Furthermore, the results indicate that WG dispersions have better performance as an adhesive compared to SPI dispersions, at least when the dispersions have been prepared at room temperature during one hour. The results in section 4.2 implied that the time for preparing the SPI dispersion affects its abilities as an

adhesive, and longer time is beneficial. In other words, the time for preparing these dispersions favors the WG dispersion. Surprisingly, Figure 11 shows that higher density has a negative effect on the internal bond value; normally a higher density results in boards with higher internal bond values [4]. However, a probable explanation might be provided by the coefficient plot in Figure 12, where a model was fitted in which the density was no longer considered as a factor but as a response. The coefficient plot implies that when SPI was employed it resulted in boards with higher density. It is also clear from Figure 11 that SPI had a negative impact on the internal bond values of the boards. Therefore, the connection between these two properties might be the explanation to why a higher density seems to have a negative impact on the IB. The higher densities when SPI was used may be a coincidence or it might be related to the tack of the glued particles; more tack means less loss of particles at the edges, which can result in boards with higher density.

Figure 12 – Coefficient plot (first component) for the density.

Regarding the thickness swelling of the boards it can be seen in Figure 13 that longer storage time of the dispersions most likely has a negative effect on the dispersions ability as an adhesive for particleboards. Once again it can be concluded that higher moisture content of the dried particles is favorable for the thickness swelling. Additionally, higher density seems to result in boards with higher TS, which is logical as higher density leaves less free space between the particles where the water can be without pressing against the particles.

Figure 13 – Coefficient plot (first and second component) for thickness swelling (TS), lower values for TS are preferable.

4.4 Effect of the gluing process - application methods 1 and 2

The experiments described in section 3.5.4 were also analyzed with software SIMCA P+. The two controlled factors were: the concentration of the wheat gluten (WG) dispersions, 12%, 16%, 20% or 24%; and the application method, application method 1 (appl. 1) or application method 2 (appl. 2). In the one step process (appl. 1) the particles are glued in one step, whereas in the two step process (appl. 2) the particles are glued in two steps with drying in between. Additionally, three factors that could not be controlled entirely, but determined, were: the density of the boards, the moisture content of the glued particles before the hot press, and the thickness of the boards. The three responses were: internal bond (IB), thickness swelling (TS), and water absorption (ABS).

It was necessary to add the interaction between the concentration and the application method as a factor, in order to obtain a model that is good enough for studying the impact of the factors on the board properties. In addition, observation WG-12_(2) was excluded, because 66.7% of the responses were missing a value. The summary of fit plot (PLS) has two components and the cumulative R2Y value is 0.74 and Q2 value is 0.61.

The scatter plot in Figure 14 illustrates the occurrence of two groups, representing the application methods. The boxes denote the experiments where application method 1 was employed and the triangles represent the experiments where application method 2 was used.

Figure 14 – The boxes in the scatter plot denote the experiments where the one step process was employed and the triangles represent the experiments where the two step process was used. Two clear groups can be seen which depend on the gluing process.

The coefficient plot in Figure 15 reveals the impact of the different factors on the internal bond value. The interaction between the concentration of the dispersion and the application method seems to be one of the factors influencing the IB value. Furthermore, it appears as if higher concentration of the dispersion is preferable, when application method 1 is employed.

In contrast, application method 2 seems to be a better option when the concentration of the dispersion is lower. Nevertheless, the application method may influence the IB regardless of the concentration; it appears as if the two step process (appl. 2) is better. Additionally, the results also show that a higher density of the boards has positive effect on the IB, which is a well known correlation [4].

Figure 15 – Coefficient plot (second component) for internal bond (IB), a high value for IB is desirable. The two last factors show the effects of the interactions between the concentration of the dispersion and the application method.

A principal factor improving the thickness swelling of the boards is the application method; it is an advantage to use the two step process (appl. 2), see Figure 16. Once again the results show that higher moisture content is beneficial regarding the TS values. However, if the moisture content is increased considerably, it will result in delamination of the boards due to steam blisters. Additionally, Figure 16 illustrates that lower density has a positive effect on thickness swelling, which is both in accordance with the other results and reasonable; since lower density means more free space between the particles where the water can be without pressing against the particles. The results show that a thicker board may have a positive impact on the TS value. There may be a connection between this factors and the moisture content of the particles. If the moisture content is higher, more steam needs to be released, which could result in thicker boards due to some springback. Generally when UMF resin is used springback normally results in boards with poorer mechanical properties [17]. However, in this case it seems like the effect of the thickness of the board contradicts the general rule. We propose that this behavior can be explained by the higher ductility of the protein-binder contra the highly cross-linked UMF resin. Increased water content can soften the protein allowing for larger deformation before fracture. Water will however, induce stresses; why an optimal water level probably exists.

Figure 16 – Coefficient plot (first component) for thickness swelling (TS), a low value for TS is preferable. The two last factors show the effects of the interactions between the concentration of the dispersion and the application method.

Similarly, application method 2 is also beneficial for the absorption property of the particleboards and so is higher moisture content of the glued particles (see Figure 17). Figure 17 shows that lower density and a thicker board results in lower ABS values. Springback usually results in boards with lower density, since the particles are pressed out towards the surface. When the boards are sanded the surfaces with higher density are removed, leaving the board with a lower total density. The same reasoning as for the affect of the thickness of the boards on the TS value can be made in this case also.

Figure 17 – Coefficient plot (first component) for absorption (ABS), a low value for ABS is preferable. The two last factors show the effects of the interactions between the concentration of the dispersion and the application method.

The proposed theory is that with application method 2, the first addition of dispersion seals the particles and, therefore, the penetration of the dispersion, added in the second step, into the wood particles is limited. As a consequence better glue bonds are obtained between the particles. Specially, for the dispersions with low concentration (low viscosity) over-penetration into the wood particles can cause poor glue bonds [19]. This is in accordance with previous studies of wheat gluten as glue for solid wood [20-21].

The application method for obtaining high IB values is dependent on the dispersion concentration. This dependence may be related to the viscosity of the dispersions; lower concentration means lower viscosity. Over-penetration of the dispersion into the wood particles tends to be a bigger problem for dispersions with low viscosity. However, dispersions with low viscosities may have an advantage, it easier to get a better distribution on the particles that might result in boards with better properties. Most likely there is less penetration when application method 2 is used; this makes it possible to use a dispersion with a lower concentration without extensive over-penetration and at the same time have improved distribution on the particles.

4.4.1 Effect of water

Depending on the employed application method the moisture may be present in different ways in the glued particles. This should be taken into account and perhaps it is a part of the explanation to why application method 2 seems to be beneficial. To elucidate this, control experiments were made with 12% WG dispersions. In one of the experiments application method 2 was employed and in the other experiment water was added to the particles, after they had been glued and dried. The results are presented in Table 8.

Table 8 – The board properties when dispersions with a concentration of 12% were used. The values are means $\pm$ standard deviation.

Sample	Thickness (mm)	Dens. (kg/m ³)	IB (MPa)	ABS (%)	TS (%)
WG-12_2 (3)	15.5 $\pm$ 0.1	633 $\pm$ 30	0.43 $\pm$ 0.09	130 $\pm$ 5	55 $\pm$ 2
WG-12_W	16.3 $\pm$ 0.3	680 $\pm$ 32	0.27 $\pm$ 0.06	122 $\pm$ 9	64 $\pm$ 3
WG-12_1 (1)*	15.6 $\pm$ 0.5	642 $\pm$ 20	0.11 $\pm$ 0.02	181 $\pm$ 5	96 $\pm$ 6
WG-12_1 (2)*	15.3 $\pm$ 0.1	714 $\pm$ 16	0.05 $\pm$ 0.03	-	-
WG-12_2 (1)*	16.5 $\pm$ 0.1	691 $\pm$ 45	0.47 $\pm$ 0.14	112 $\pm$ 8	57 $\pm$ 3
WG-12_2 (2)*	16.7 $\pm$ 0.1	630 $\pm$ 14	0.35 $\pm$ 0.06	121 $\pm$ 1	52 $\pm$ 3

The first number in the sample name points to the concentration (12 %) of the dispersion and then the application method (appl.1, appl. 2 or addition of water in a second step) is indicated. When several boards were made with the same recipe, the numbers in the brackets distinguishes them from each other.

*These boards were made earlier, described in section 3.5.4. However, it is still interesting to compare the board properties.

The control experiments with the 12% WG dispersions indicate that better results are obtained if water is added in a second step compared to employing application method 1. Nevertheless, the two step process (appl. 2) induces considerably better performance. Most likely, the presence of moisture and how it is available is an important factor for the performance of the protein-based adhesives. The balance between the negative effects of water (steam blisters) and the positive effect (softening of protein) is not known at present.

5. Conclusions

The major conclusion from this study is that wheat gluten (WG) and soy protein isolate (SPI) can be used as binders for particleboard. However, to expose the proteins best possible properties for this application, they have to be processed. The results from this study reveal that it is definitely an advantage to disperse the proteins compared to employing them as dry protein powders. Furthermore, the results indicate that it is not beneficial to store the dispersions for more than one day.

According to this study, it is a drawback to use green particles instead of particles that have been previously dried; although, the glued particles are dried to approximately the same moisture content. One possible explanation is that the present water is bound in different ways. Additionally, an important parameter to consider is that the properties of the particles can depend on its composition, how and for how long it has been stored.

Furthermore, when preparing WG dispersions it appears as if lower temperature for the preparation of the dispersions is an advantage, while the time seems to be a trivial factor, for the investigated interval. On the other hand, for SPI dispersions it appears as if longer time to prepare the dispersion is beneficial, whereas the temperature seems to be of no significant importance, for the investigated interval.

The overall results suggest that better board properties are obtained if the moisture content of the glued particles is higher. Nevertheless, this is only true in the investigated interval, if the moisture content would be much higher the boards would most likely delaminate due to steam blisters.

Based on the results for the experiments conducted with WG dispersion it seems like it is preferable to use the two step process (appl. 2) to glue the particles, compared to the one step process (appl. 1). When looking at the internal bond property it appears as if the interaction between dispersion concentration and the application method is a significant factor. When dispersions with lower concentration are employed it seems to be beneficial to glue the particles according to application method 2. On the other hand application method 1 may be preferable when the dispersion has a higher concentration.

6. Future work

The present study reveals that the gluing process of the particles is of significant importance for the performance of wheat gluten (WG) dispersion as an adhesive for particleboards. Furthermore, it seems like employing the two step process is an advantage compared to using the one step process. Therefore, further studies will be performed to obtain a better understanding of this behavior. Different microscopic methods will be tested to investigate the penetration of the dispersion into the wood particles and the distribution of the dispersion on the particles.

The utilization of cross-linker will be explored in order to create adhesives with improved performance as a binder for particleboards. The idea is also to investigate the usage of the cross-linker directly in the WG dispersion, and to add it separately to the particles in the second step.

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