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Use of Industrial Hemp Fibers to Reinforce Wheat Gluten Plastics

C. Wretfors · S.-W. Cho · M. S. Hedenqvist ·
S. Marttila · S. Nimmermark · E. Johansson

Abstract The next generation of manufactured products must be sustainable and industrially eco-efficient, making materials derived from plants an alternative of particular interest. Wheat gluten (WG) is an interesting plant material to be used for production of plastic similar materials due to its film-forming properties. For usage of plastics in a wider range of applications, composite materials with improved mechanical properties are demanded. The present study investigates the possibilities of reinforcing WG plastics with hemp fibers. Samples were manufactured using compression molding (130 °C, 1600 bar, 5 min). Variation in fiber length, content (5, 10, 15 and 20 wt%) and quality (poor, standard, good) were evaluated. Mechanical properties and structure of materials were examined using tensile testing, light and scanning electron microscopy. Hemp fiber reinforcement of gluten plastics significantly influenced the mechanical properties of the material. Short hemp fibers processed in a high speed grinder were more homogenously spread in the material than long unprocessed fibers. Fiber content in the material showed a significant positive correlation with tensile strength and Young's modulus, and a negative correlation with fracture strain and strain at maximum stress. Quality of the hemp fibers did not play any significant role for tensile strength and strain, but the Young's modulus was significantly and positively correlated with hemp fiber quality. Despite the use of short hemp fibers, the reinforced gluten material still showed uneven mechanical properties within the material, a result from clustering of the fibers and too poor bonding between fibers and gluten material. Both these problems have to be resolved before reinforcement of gluten plastics by industrial hemp fibers is applicable on an industrial scale.

Keywords Composite materials · Hemp · Plastics · Renewable raw materials · Wheat gluten

C. Wretfors · E. Johansson (✉)
Faculty of Landscape Planning, Horticulture and Agricultural
Sciences, Swedish University of Agricultural Sciences,
P.O. Box 104, 230 53 Alnarp, Sweden
e-mail: Eva.johansson@ltj.slu.se

S.-W. Cho · M. S. Hedenqvist
Fibre and Polymer Technology, Royal Institute of Technology,
100 44 Stockholm, Sweden

S. Marttila
Faculty of Landscape Planning, Horticulture and Agricultural
Sciences, Swedish University of Agricultural Sciences,
P.O. Box 102, 230 53 Alnarp, Sweden

S. Nimmermark
Faculty of Landscape Planning, Horticulture and Agricultural
Sciences, Swedish University of Agricultural Sciences,
P.O. Box 105, 230 53 Alnarp, Sweden

Introduction

There is currently worldwide interest in high performance bio-based plastics and composite materials due to increasing demands for environmentally friendly materials for industrial use [1] and to the depletion of petroleum resources.

Wheat gluten-based bioplastics are an interesting alternative to traditional synthetic plastics in various applications due to their combination of mechanical, oxygen barrier and film-forming properties [2–4]. Wheat gluten (WG) plastic material can be manufactured using several processing methods, including extrusion, compression molding and solution casting [5]. Compression molding and extrusion are commercially more interesting due to

faster processing times. During compression molding, high temperature, plasticizer content and type, pH, processing time and shear rate all influence the mechanical properties due to their importance for protein polymerization [6, 7]. Sulphydryl from the amino acid cysteine is responsible for creating disulphide cross-links during oxidation. The reorganization of the intramolecular disulphide bonds into intermolecular disulphide bonds is an important part of the aggregation process [8, 9]. The processing window is determined by depolymerization and/or an over extensive aggregation of the proteins, both events leading to a decrease in mechanical properties [10].

One possibility for expanding the potential areas of application for bio-based plastics is to improve mechanical properties, such as tensile and impact strength, e.g. by reinforcement with bio-fibers. Plants containing fibers of interest for the manufacture of engineering materials include flax, hemp, jute, coconut and nettles [11]. The main advantages of using plant fiber are renewability, high strength and elastic modulus, low density, non-abrasiveness and biodegradability [12]. Engineered wood and lignocellulosic composite technologies can lead to considerable addition of value to a diverse number of raw materials. The development of industrial composite processing technology will also automatically provide producers with the possibility to adapt to the constantly changing qualities of raw materials. At present, the level of performance of engineered composite products limits the application of such materials [1].

The use of plant fibers to reinforce plastics has been investigated in quite a number of studies (e.g. [11]). However, the use of plant fibers for reinforcement of bioplastics is still relatively uncommon and primarily uses different types of plant fibers in soy-based bioplastics [13, 14]. Some recent studies investigated the possibilities of using natural fibers to reinforce plasticized WG [15, 16]. The quality of natural fibers for reinforcement of plastics is influenced by a number of factors including plant variety, growing climate, harvest time, maturity, retting, methods of decortication and other technical processes [17]. All these variables combine to create one of the main drawbacks encountered when trying to design new materials based on plant fibers, namely the large variation in fiber properties such as tensile strength and surface geometry [12]. This variation exists among fibers from plants grown in the same plot, and even within groups of fibers from the same plant [18]. The general advantages of using plant fiber instead of synthetic fiber to reinforce bio-materials are, however, the creation of a totally "green" material being able to characterize as fully renewable.

The main aim of the present study was to investigate the possibility of using industrial hemp fibers to reinforce WG plastics manufactured by compression molding. An additional aim was to study the influence of the amount of fiber added and the quality of the fiber on tensile strength, strain and Young's modulus (E). Furthermore, fiber distribution and fracture surfaces were investigated using stereo light microscopy and scanning electron microscopy (SEM).

Materials and Methods

Materials

WG powder was supplied by Reppe AB, Lidköping, Sweden. According to the provider, the powder consisted of 84.8 wt% wheat gluten proteins, 8.1 wt% wheat starch, 5 wt% water, 1.34 wt% fat and 0.76 wt% ash. Glycerol with a concentration of at least 99.5 wt% and a water content of less than 0.5 wt%, was supplied by Karlshamns Tefac AB, Karlshamn, Sweden.

Three different types of industrial hemp fibers were selected in order to provide three distinct quality levels:

- Hemp fiber type 1—poor quality: Unretted hemp stalks, harvested by forage harvester, were partially dried in plastic tubs with fans placed underneath the hemp material. The resulting fiber was heavily retted and designated 'poor' quality.
- Hemp fiber type 2—standard quality: Unretted hemp stalks, harvested by forage harvester, were processed through a hammer mill with sieve size 50 mm (Kamas Industri AB, Sweden) and then through a step cleaner (Hergeth Gmbh, Germany) to remove larger size shives. The resulting fiber type was designated 'standard' quality.
- Hemp fiber type 3—good quality: Unretted hemp stalks, harvested by forage harvester, were processed through a hammer mill (Kamas Industri AB) without any sieve installed and then through a step cleaner (Hergeth Gmbh, Germany) to remove large and small shives. Because no sieve was used, the fiber passed quickly through the mill, resulting in less fiber damage. The resulting fiber type was designated 'good' quality.

Sample Preparation for Compression Molding

Dough was prepared by mixing gluten and glycerol to a content of 30 wt% (given as the mass of glycerol per total weight of glycerol and WG). Each blend was mortared for 5 min at about 150 rpm using a Mortar Agate from VWR International. When the dough was assessed as being homogeneous, hemp fiber of the desired type and amount was added.

For the initial tests, 5, 10 and 15% of poor, standard and good quality hemp fiber were added to form separate

samples of hemp fiber-reinforced gluten plastics. The samples were compression molded directly after addition of the hemp fibers, following the methodology described below. During these initial tests, it was found that the fibers were too long, resulting in poor fiber distribution throughout the plastic.

For all subsequent samples, shorter fibers were used. These were prepared by removing any woody substances manually and then processing the fibers in a high speed grinder (model A10, IKA-WERKE, Staufen, Germany) for 2×10 s, resulting in very short fibers to facilitate a more uniform distribution through the plastic material. The gluten dough together with the desired amount (5, 10, 15 and 20%) of short industrial hemp fibers were added to an electrical blender, i.e. a type of regular food mixer of large size (Waring Commercial, USA), and mixed until an even blend of dough and fibers was achieved.

Compression Molding

Compression molded films were processed using a PHI press (Pasadena Hydraulics Inc, California, USA). Portions of 10 g of dough were placed in an aluminum frame between Mylar foils, which in turn were placed between metal plates. The frame was used to obtain square films with sides of 100 mm length and a thickness of 0.5 mm. The molding temperature was 130 °C, and the pressure was set to 100 bar, which gave an applied pressure of 1600 bar. The molding time was 5 min. After molding, the plates were removed from the press and the films were allowed to cool to ambient temperature. The Mylar foils were then removed and the films were separated from the frame using a scalpel.

Sample Thickness Measurements

The thickness of each sample was measured using a Mitutoyo IDC-112B micrometer (Mitutoyo Scandinavia AB) in accordance with SCAN-P 7:96 at 23 °C and 50% relative humidity (RH), at a static pressure of 100 kPa [19].

Tensile Testing

A Zwick Z010 tensile strength tester (ZwickRoell) equipped with a 500 N load cell controlled by a testXpert 7.1 (Lambda Instruments AB) was used to determine the mechanical properties of the samples. The measurements were performed according to ISO 527-3:1995(E) [20]. Dumbbell-shaped specimens were punched out with a narrow width of 4 mm and conditioned for 3 days at 23 °C at 50% RH before testing. A crosshead speed of 100 mm/min and an initial grip distance of 40 mm were used. The tensile strength was calculated based on the original

cross-section in narrow part. Fifteen replicates of each sample were tested.

Light Microscopy (LM)

Samples from each treatment were studied by stereo light microscopy with a digital camera Leica DC 300 (Leica Microscopy Systems Ltd, Cambridge, UK) in order to evaluate the fiber distribution throughout the plastic. Nine images were taken for each sample in order to cover the entire sample area.

Scanning Electron Microscopy (SEM)

An LEO 435VP scanning electron microscope (Cambridge, UK) with a secondary electron detector at acceleration voltage of 10 kV was used to study the fiber raw material and the finished plastic material. The native samples were mounted on the stubs and sputtered with an Au/Pd 3:2 coating (JFC-1100, JEOL, Tokyo, Japan).

Images were taken of the fiber raw material to observe the state of the fiber bundle surfaces in the three fiber quality types.

Plastics samples of the specific treatment that showed the biggest variation in Young's modulus during tensile testing were chosen for further SEM analyses. The fracture surface of samples from this treatment was compared with that of another almost similar treatment that exhibited a much smaller variation in Young's modulus, the only difference being the fiber quality. The surface of the samples in the area of the actual fracture was observed in order to study the fiber fracture and distribution.

Statistical Analysis

Statistical analysis was carried out using the SAS software package for Microsoft Windows (SAS Institute Inc, NC, USA). Analysis of variance (ANOVA) was carried out, followed by calculation of means for the different treatments with significance determined using LSD (0.05).

Results

Fiber Quality

The good quality fibers showed thick fiber bundles with relatively clean and only mildly damaged fiber surfaces when studied using SEM (Fig. 1a). The poor quality fibers sustained a larger degree of surface damage and fiber degradation, with thinner fiber bundles (Fig. 1b). The standard quality fibers were relatively undamaged and clean, with a mixture of thinner and thicker fiber bundles.
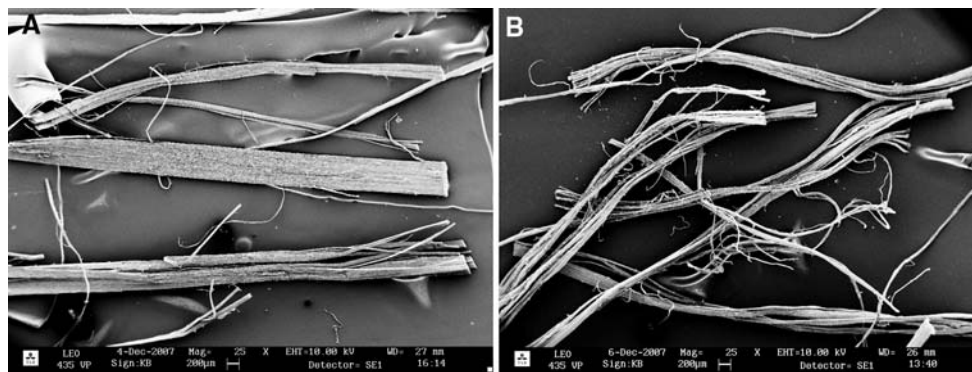
Fig. 1 SEM images showing short hemp fibers of **a** good and **b** poor quality. The scale bar corresponds to 200 μm

Films With Long Fibers

The compression molded films containing the long fibers were very non-homogeneous, regardless of amount and quality of fiber, and the fibers were very unevenly distributed within the material. Fibers tended to be lumped together in bundles (results not shown). As the results were not satisfactory, although promising, further work including tensile testing was carried out on samples to which short fibers were added.
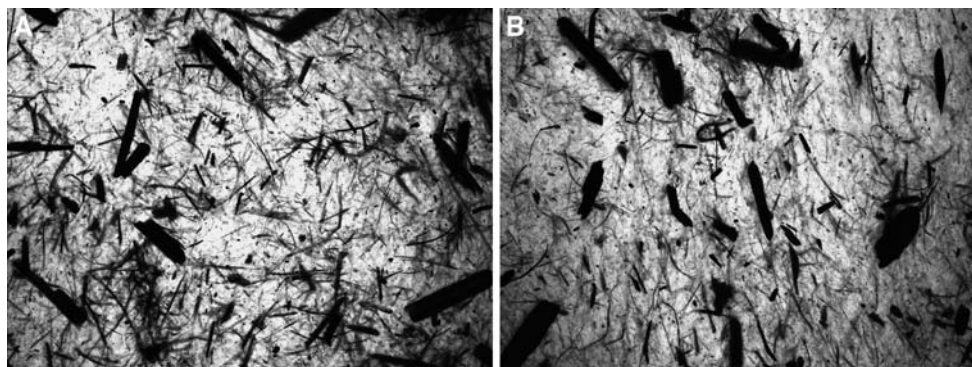
Films With Short Fibers

When the fibers added were short instead of long, a much better distribution of fibers throughout the gluten matrix was observed. However, also the short fibers were clustered

together in bundles and an uneven distribution of also these fibers was observed (Fig. 2a, b). The fibers had a random orientation in the matrix. Some contaminating plant tissue was also seen. No clear difference in the behavior of the different fiber qualities in the gluten film was seen by light microscopy.

Tensile Testing of Short Hemp Fiber-reinforced Gluten Plastics

Tensile strength and Young's modulus of the short hemp fiber-reinforced gluten plastic samples varied significantly in relation to hemp fiber content and quality (Table 1). However, standard deviations for measured parameters were generally large in the fiber-reinforced gluten materials. Standard deviation for Young's modulus and fracture

Fig. 2 Light microscopy images showing gluten films reinforced with short hemp fibers of **a** good and **b** poor quality

Table 1 Mean squares from analyses of variance (ANOVA) of treatments (content and quality of short industrial hemp fibers in reinforced gluten plastics) on tensile test parameters

Source	DF	Maximum stress (MPa)	Strain at maximum stress (%)	Fracture stress (MPa)	Fracture strain (%)	Young's modulus (MPa)
Treatment	12	13.9***	21.7***	5.0***	21.2***	65.9***
Error	182	1.8	0.1	1.3	0.1	4.8

*** Significant differences were found

Table 2 Mean E modulus and strain at fracture of hemp fiber reinforced gluten plastics containing different fiber amounts and qualities

Treatment	E-modulus (MPa)	Fracture strain (%)
Wheat gluten	23.7 (7.1)	149.2 (15.6)
5% fiber, poor	40.6 (13.2)	42.0 (15.7)
10% fiber, poor	84.2 (53.1)	21.6 (9.7)
15% fiber, poor	108.9 (59.0)	12.6 (4.2)
20% fiber, poor	141.9 (52.8)	10.2 (2.3)
5% fiber, average	45.6 (18.5)	37.3 (14.9)
10% fiber, average	88.1 (56.4)	18.3 (8.4)
15% fiber, average	113.2 (65.0)	12.8 (3.9)
20% fiber, average	228.9 (116.1)	9.2 (4.1)
5% fiber, good	41.4 (14.3)	45.9 (17.4)
10% fiber, good	82.0 (41.3)	21.7 (12.9)
15% fiber, good	143.1 (145.5)	15.8 (9.1)
20% fiber, good	227.1 (93.5)	8.6 (2.2)

$N = 15$. Standard deviation is shown within parenthesis

strain was in average 52.3 and 40.0%, respectively, in the reinforced materials as compared with 30.0 and 10.5%, respectively, in the pure gluten samples (Table 2). The spread results of the mechanical properties of reinforced samples are most likely due to the uneven distribution of fibers in the materials.

Gluten plastics without any hemp fiber added were found to have low tensile strength (maximum stress and fracture stress) and Young's modulus. Mean values of maximum stress and Young's modulus were 2.6 and 23.7 MPa, respectively, in the gluten samples. Strain at maximum stress and fracture strain were found to be relatively high, 147.3 and 149.2%, respectively, in the gluten samples

(Table 3). With increasing content of short hemp fibers tensile strength and Young's modulus increased significantly. The 20% fiber content led to a doubled of the maximum stress (4.7 MPa) and an almost 10-fold increase in Young's modulus (199.3 MPa). The strain decreased with an increasing content of short hemp fibers; 20% fiber content lead to a 20-fold decrease in strain (strain at maximum stress was 6.1% at 20% fiber content and 147% when no fibers were added; Table 3). No significant influences of fiber quality on tensile strength or stress were found. However, Young's modulus was found to increase significantly with improved fiber quality, from 94.0 MPa for bad fiber quality to 123.4 MPa for good fiber quality (Table 4).

Fracture Surfaces of Short Industrial Hemp Fiber-Reinforced Gluten Plastics

SEM analysis showed that the fracture surface of gluten film without fiber reinforcement was very uniform (Fig. 3). The gluten film tended to break in a sheet-like manner, similarly to proteins in the starchy endosperm of dehydrated cereal grains. Samples from the treatment with 15% good quality fiber were chosen for SEM analysis since these samples showed a large variation in tensile strength measurements. The fracture surfaces of these samples were compared with the fracture surfaces of samples of the treatment with 15% poor quality fibers, for which a much smaller variation in tensile testing was. The SEM images showed that the fibers were unevenly distributed throughout the material (Fig. 4), as were found by light microscopy (Fig. 2). Figure 4a shows uneven fracture surfaces in films with good quality fibers, while Fig. 4c presents the

**Table 3** Mean values of tensile strength measurements at different fiber content

Fiber content (%)	Maximum stress (MPa)	Strain at maximum stress (%)	Fracture stress (MPa)	Fracture strain (%)	E-modulus (MPa)
0	2.6cd	147.3a	2.5bcd	149.2a	23.7d
5	2.3d	35.9b	1.9d	41.5b	42.5d
10	3.3c	13.8c	2.5c	20.5c	84.8c
15	4.1b	10.0d	2.9ab	13.7d	121.7b
20	4.7a	6.1d	3.3a	9.3e	199.3a

Means with the same letters within a column do not differ significantly (LSD 0.05)

Table 4 Mean values of tensile strength measurements at different fiber quality

Fiber quality	Maximum stress (MPa)	Strain at maximum stress (%)	Fracture stress (MPa)	Fracture strain (%)	E-modulus (MPa)
Bad	3.5a	16.8a	2.6a	21.6a	94.0b
Average	3.8a	15.3a	2.9a	19.3b	118.9ab
Good	3.5a	18.0a	2.6a	22.9a	123.4a

Means with the same letters within a column do not differ significantly (LSD 0.05)
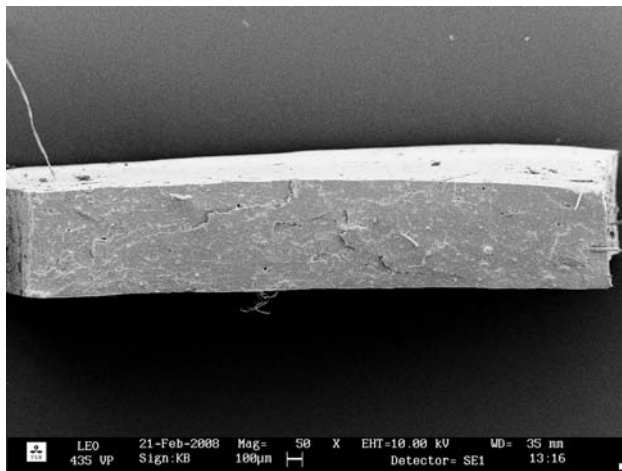
Fig. 3 SEM image showing gluten plastic film without fiber reinforcement. The scale bar corresponds to 100 μm

corresponding fracture surfaces for poor quality fibers. In both cases, fracturing seemed to have started around areas where the fibers were clustered and where air pockets had started to form, and the fibers were protruding from the matrix. The more even fracture surfaces (Fig. 4b, d) show clearly that gluten film had broken in a sheet-like manner, as in Fig. 3. Fewer fibers were protruding from the matrix than in the uneven fracture surfaces, but there were still air

pockets near the clustered fibers. When the protruding ends of fibers were examined more closely, it was seen that the fibers had been pulled out of the gluten plastic matrix, rather than breaking during tensile testing (Fig. 5). The typical blunt end of pulled-out fiber bundles shown in Fig. 5a suggests that this is a surface formed during milling of the fibers. For comparison, Fig. 5b shows the only fiber end we could find in the material screened where the fiber appeared to have actually broken during tensile testing, as shown by the serrated edges of the fiber bundle.

Discussion

The present study confirmed the finding by Kunanopparat et al. [15, 16] that it is possible to use industrial hemp fibers to reinforce wheat gluten plastics. In contrast to the study by Kunanopparat et al. [15], both long and short industrial hemp fibers were tested in the present investigation, and short ones were found to give a better distribution of fibers in the gluten matrix. The advantage of using short industrial hemp fibers instead of long has also been pointed out by other authors, e.g. Mohanty et al. [14] for reinforcing soy protein plastics.

Gluten films without additives or reinforcements, processed by compression molding are known to have low
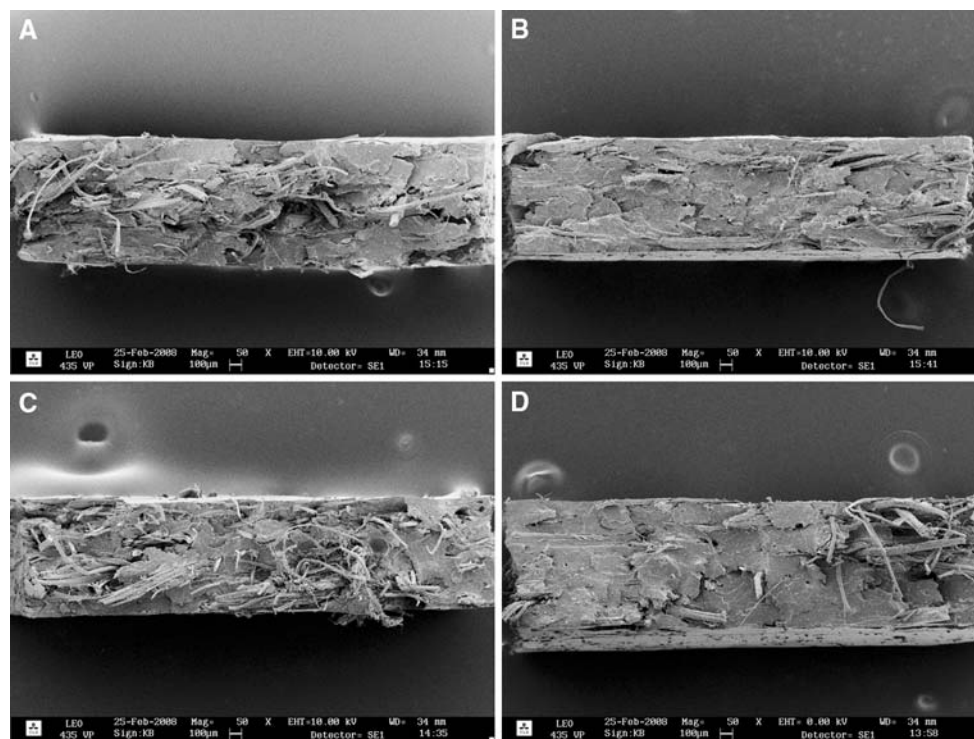
Fig. 4 SEM images showing fracture surfaces of gluten film reinforced with short hemp fibers of **a** good quality fibers and uneven fracture surface, **b** good quality fibers and more even fracture surface, **c** poor quality fibers and uneven fracture surface, and **d** poor quality fibers and even surface. The scale corresponds to 100 μm
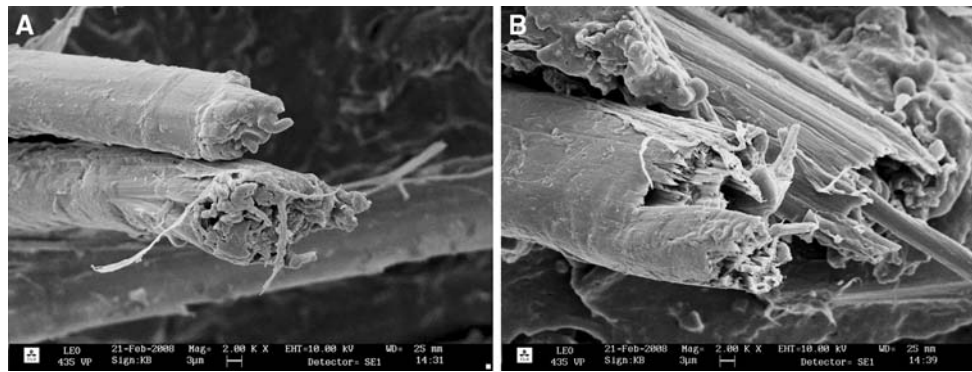
Fig. 5 SEM images showing magnification of pulled-out fibers from a fracture surface, **a** typical blunt-end fiber and **b** more rare broken fiber with serrated edges. The scale bar corresponds to 3 μm

tensile strength and Young's modulus [10]. In the present study, both tensile strength and Young's modulus of wheat gluten plastics were increased significantly (doubled and 10-fold, respectively) by reinforcement with hemp fibers, as was also found by Kunanopparat et al. [15]. Composites built on gluten material have also been evaluated by other authors using e.g. silica, hydroxyethyl cellulose and methylcellulose as microfibers, also doubling the tensile strength of the pure gluten material [21–23]. Addition of natural fibers has also been found to increase the tensile properties of a range of other materials (e.g. [11, 13, 14, 24]). Also, the increase of tensile strength and Young's modulus in those studies has been found to increase up to 2.5-fold for tensile strength and up to 4-fold for Young's modulus [11, 13, 14, 24]. However, by the use of soy protein as the matrix and a combination of twin-screw extrusion and injection molding for producing the material, the pure soy film showed substantially higher tensile strength properties [11, 13, 14, 24] as compared to the pure gluten films in our study. Thus, in order to be able to produce industrially interesting alternatives of hemp-reinforced gluten plastic materials, additives and treatments leading to higher polymerization of the gluten proteins has to be taken into considerations as well. Examples of such additives and treatments are e.g. additions of NaOH and/or salicylic acid increasing polymerization and usefulness of gluten plastics [5, 25], and increased processing temperature [10]. Increased processing temperature has also been shown to be a useful treatment as regards to production of composite materials from gluten and hemp fibers [16].

In other studies, natural fibers have been shown to compare favorably with glass fibers, and hemp fibers have been shown to out-perform e.g. kenaf fibers [26]. As in other investigations [13], fiber content in the present study had a significant effect on tensile strength, with an increase in fiber content resulting in a stronger and stiffer material. Contrary to previous results [13], our investigation did not show a negative influence of increasing fiber content on

fiber dispersion. The reason might be the different plastic and fiber materials used in the studies, soy protein and pineapple leaf fibers in that by Liu et al. [13] compared with gluten proteins and hemp fibers in our study. Also, the differences in production methods of the materials might have been of relevance, e.g. twin-screw extrusion and injection molding [13] compared to compression molding in the present one. If a stronger gluten protein material with higher stiffness is required, it is desirable to include as much short industrial hemp fiber reinforcement as possible.

Although the short industrial hemp fibers were better distributed in the gluten matrix than the long ones, a relatively high standard deviation was still found during the tensile testing and this was most likely caused by an uneven distribution of the fibers throughout the plastic. The fibers showed a tendency to cluster together during the mixing process and this caused weaker, unreinforced zones in the material, as could be seen clearly by both light microscopy and SEM. Uneven distribution of natural fibers in plastic matrices has also been reported in previous studies [13, 24]. In order to improve the distribution of fibers in the matrix a number of solutions can be adopted. First, the blender used for this experiment did not fully succeed to create an evenly mixed material. Thus, solutions could be to use a high performance mixer, leading to a more intensive mixing under a set temperature, or to adopt the method used by Liu et al. [13, 24] combining twin-screw extrusion and injection molding. Other possibilities are addition of substances that increase the viscosity of the plastic dough or decrease fiber size even further into micro or nano scales.

The present investigation showed limited influences of fiber quality on tensile strength properties, with significant effects only being recorded for Young's modulus. To our knowledge, there are few previous investigations on the relationships between fiber quality and tensile strength properties of natural fibers. The published studies investigating the possibility of reinforcing gluten plastics with

industrial hemp fibers did not compare quality of the hemp fibers and relationships to tensile properties [15, 16]. As to the results of the present study, more even distribution of hemp fibers in the gluten matrix is needed before influence of fiber quality on tensile properties can be confirmed.

The limited effect of fiber quality on tensile strength properties was probably due to the poor bonding between fibers and plastic observed in the present study. This poor bonding was visible as a pull out effect, where the fibers did not fracture during tensile testing. Air pockets were also observed around clustered fibers, again suggesting poor bonding between the fibers and the matrix. Due to the fact that wheat gluten plastic itself is much weaker than any type of hemp fibers, breaks always took place within the gluten matrix and the fibers were not broken at tensile testing. If a stronger bonding had been achieved between the fibers and the plastic, the different fiber qualities might have had a more dramatic effect. Poor bonding between natural fibers and plastic materials has also been reported by other authors [13, 24]. Different solutions have been suggested, e.g. addition of alkali solutions to decrease the interfibrillar region by removing the hemicellulose and lignin [24]. Other suggested solutions are additions of compatibilizers such as polyester amide grafted glycidyl methacrylate [13]. Treatment of Indian grass fibers with alkali solution has been shown to lead to a more homogeneous dispersion of fibers in plastics and also to an improvement in fiber reinforcement efficiency [24], while addition of compatibilizer to soy-based bioplastic reinforced with pineapple leaf fibers led to increased tensile and flexibility properties and impact strength, as well as better dispersion of the fibers [13]. For better and more even distribution of short industrial hemp fibers within gluten protein plastics, a method is needed in which a cross-linking/coupling/compatibilizer agent can be added to help create hydrogen bonds to both the fibers and the plastic.

Conclusions

Short industrial hemp fibers might in the future be a good solution for reinforcement of gluten plastics. Hemp fibers seem to have the potential of creating a strong, stiff and sustainable eco-efficient gluten based composite material for the automotive and building product industry. Before the material can be used, however, problems with uneven distribution of hemp fibers and poor bonding between fibers and the matrix in the material have to be resolved. Possible solutions for a better distribution of fibers in the material include use of a extrusion or intensive mixing at a set temperature, increased viscosity of the plastic dough, an even further decrease of the hemp fibers into micro or nano sizes and/or additions of cross-linking substances for creating linkages between fibers and the dough.

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