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Processing and properties of sorghum stem fragment-polyethylene composites

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Abstract
Composites prepared with whole sorghum stem fragments reinforcing a polyethylene matrix were studied using ten different sorghum genotypes. Using a robust processing protocol, it is shown that for a given sorghum genotype, the composition of the stem fragments varies depending on the size of the sieved fragments but with the genotype effect being larger than the sieving effect. There is a variation of mechanical properties between the genotypes (from 0.6 to 1 GPa for modulus, from 7.2 to 11.5 MPa for tensile strength and from 4.4 to 6.2 kJ/m2 for impact strength). The genotypes giving the best tensile mechanical properties are the ones which have the highest viscosity, which show during blending the largest energy dissipation and which have the less decrease of size after processing. There is a weak correlation between tensile mechanical properties and resistance to impact suggesting that it is not the same tissues or physical properties which contribute to these two tests.

Keywords: Biocomposites; Sorghum; Mechanical properties; Injection molding

1 Introduction

Sorghum is a low input and multi-purpose crop which has traditionally been used in two main sectors: either directly as food for human being and feed in Africa and Asia or mainly as animal feeds in the developed countries and in Latin America. High environmental adaptation, high productivity and tolerance to salt and drought are among the properties of sorghum. This gives characteristics which are of great interests in the view of a possible climate change in Europe. Because sorghum stems are rich in soluble sugars (i.e. glucose, sucrose and fructose) and insoluble carbohydrates (i.e. cellulose and hemicellulose), they have been considered as energy crop parts for producing biofuels, bioenergy, biogas and bioethanol (Almodares and Hadi, 2009; Matsakas and Christakopoulos, 2013; Nikzad et al., 2014; Ostovareh et al., 2015 ,). A few value-added products have been investigated like cellulose pulp for the production of paper (Albert et al., 2011; Belayachi and Delmas, 1995; Gençer and Şahin, 2015; Khristova and Gabir, 1990), particleboard (Khazaeian et al., 2015), chemicals and other bio-products (Dong et al., 2013; Tanamool et al., 2013), SiC nanoscale particles and nanorods from burned leaves (Qadri Sert al., 2013) and reinforcement for fly ash-based geopolymer (Chen et al., 2013). However, the sorghum stalks/stems have not yet been thoroughly investigated as a renewable natural resource for non-food applications.
Very few studies reported the use of sorghum stems in polymer composites. Thermoplastic composite panels were prepared with high-density polyethylene (HDPE) by hot-pressing layers of sorghum stalks and HDPE films (Qi et al., 2013). Poly(L-lactide) composites reinforced with sweet sorghum fiber residue obtained after sugar extraction of sorghum stalks were studied (Zhong et al., 2010). However, regarding the qualities of sorghum, it is worth investigating the possibility to use stem fragments to reinforce polymers. It is the first objective of the research reported here. It has to be said that the whole plant stem is broken into elongated pieces (called fragments in this paper) of dimensions inferior to millimeters by mechanical means (i.e. cutting and milling or grinding). No isolated fibers are extracted from the sorghum stems. The second objective is to

select appropriate processing conditions in a protocol which is able to highlight the influence of the filler on the mechanical properties of the composite. It aims to study the relationships between genotype characteristics and composite properties, since sorghum genotypes present a large chemical composition variability. Contrary to most plant fillers which consist of extracted fibers with a high cellulose content, the use of biomass fragments obtained by grinding whole stems is posing additional challenges, but offers the opportunity to avoid chemical treatments and to decrease cost. How the biochemical composition of the plant stem or its histological structure are influencing the breaking of the stem, its mechanical resistance, and the type of tissues exposed at the stem fragment surface, for example, is unknown. Such aspects are critical since they will control the final properties of the composite.

There have been numerous published works dealing with the understanding the influence of the structure of plant parts able to be used in non-food applications on final properties, as for example for flax, a fiber used for reinforcing polymers (Bourmaud et al., 2013; Thuault et al., 2015). When using plants to reinforce polymers, the situation is complicated by the fact that in most cases, the reinforcing material (i.e. fibers) are extracted from the plant, as in the case of flax, hemp or curauá. These extracted materials are mostly composed of cellulose, not representative of the whole plant stem. There is currently no data on the relation between the whole plant properties and the properties of composites prepared with these plants considering different genotypes from one given plant species. One of the reasons could be the difficulty to prepare in a very robust manner composites using small amount of plant materials. To the best of our knowledge, aside a similar work on miscanthus from our teams (Girones et al., 2016), there is no information about the direct relationship between the histological structure and biochemical composition of the whole stem plant and the mechanical properties of the manufactured polymer composites, considering only one plant species and its various genotypes. This article is a step towards this goal.

After removing grains, leaves and leaf sheaths, dried sorghum stems from ten different genotypes were milled in controlled and reproducible conditions to produce elongated stem fragments. Fragments with controlled size distribution were selected by sieving and used to prepare composites. A robust method for preparing composites and testing their mechanical properties was devised in order to ensure that any change in the mechanical performance of a composite prepared with a given composite was only due to the influence of the variability of the sorghum stem fragments.

2 Materials and method

2.1 Preparation of reinforcement stem fragments

First, 396 sorghum genotypes were screened for their stem biochemical composition and ten genotypes were selected in order to maximize the coverage of the variability of stem biochemical properties.

These ten genotypes of sorghum were harvested and dried in October 2013 by Eurosorgho (France), which provided stem sections from 30 cm to 1 m length. Some of their stem phenotypic characteristics are presented in Table 1. The heritabilities of the different components of the biomass are quite high, underlying the fact that the differences observed between genotypes are quite stable even when they are exposed to different environmental conditions (Trouche et al., 2014). After reception, the stems were stored in a closed shelter to protect them from rain and direct sunlight. In order to be used as polymer fillers, stems had to be mechanically transformed to elongated fragments with a mean particle size in the order of 500 µm by first removing leaves and leaf sheath residues. Dried stem sorghum were then cut into smaller pieces of about 20 mm length by using a garden pruner. These 20-mm stem pieces were then ground in a Hellweg M50/80 granulator (Germany) designed for plastic pelletization and equipped with a 2.5 mm sieve. To ease the next milling step, the cut pieces were mildly dried in a Binder circulation air oven (FED line, Germany) at 60°C for at least 5 h. Size was further reduced by a coffee mill (Carrefour home, France) for 40 sec, with intervals of 10 sec separated by 5 sec pauses to avoid overheating of the mill, giving stem fragments with a wide size distribution. Sieving was carried out to narrow the distribution and to ensure a homogeneous fragment size. Stem fragments were sieved in a Retsch AS200 Digit shaker (Retsch, Germany), allowing separation into fractions from 100 to 1000 µm. Sieving was conducted in two steps on 20 g samples with the shaker operating at 50 mm amplitude (2.5 mm/g) for 8 minutes and then at 40 mm amplitude (2 mm/g) for 5 min. Sieves with open pore sizes of 1000, 600, 400, 300, 200 and 100 µm were used. The fraction collected between the 300 µm and 200 µm sieves, with aspect ratio (length/diameter) about 5.5 was used to prepare the compounds with the polymer. Prior to compounding, fragments were dried overnight in an air-circulating oven at 60°C to remove moisture.

2.2 Polymer matrix and coupling agents

Since the sorghum fragments have a low temperature resistance (Bakeer et al., 2013), a low density polyethylene with a low melting point (LDPE) produced by Sabic® (LDPE 1965T, Sabic Europe, melt index 65g/10min (190°C/2.16 kg), melting point 104°C, density 0.919 g/cm^3, tensile strength at break 7 MPa) was selected as the polymer matrix. Orevac® 18507, (Arkema, France, melt index 5g/10min (190°C/2.16 kg), melting point 128°C, density 0.954 g/cm^3, tensile strength at break 10 MPa), a high density polyethylene with a high content of maleic anhydride (MA-g-PE), was used as the coupling agent in a proportion of 5 wt% on dry fragment basis in order to improve the mechanical properties of composites and also enhance any possible differences caused by the different genotypes

2.3 Preparation of composites

Composite blends comprising 30 wt% by mass of sorghum stem were prepared in a two roller counter rotating Haake™ Rheomix intensive kinetic lab mixer (Haake Polylab OS torque rheometer, Thermo Scientific, USA) having a mixing chamber volume of 50 cm^3. The mixer was set to operate at 60 rpm for 9 minutes with the temperature set at 150°C to prevent thermal degradation of sorghum fragments. MA-g-PE (5 wt% on dry fragment basis or 1.5 wt% of the composite) was added to the mix once the reinforcing elements was well dispersed in order to minimize hydrolysis of the maleic anhydride groups and to limit stem fragments attrition. The compounding of the three components (polymer matrix, stem fragments and coupling agent) was performed in four steps to avoid clogging and obtain the best homogeneous distribution of sorghum stem fragments inside the matrix:

- Step 1 (t = 0 min): addition of 37.5 wt% of LDPE.
- Step 2 (t = 1 min): addition of 15 wt% of sorghum stem fragment and 12.5 wt% of LDPE.
- Step 3 (t = 3 min): addition of 15 wt% of sorghum stem fragment and 12.5 wt% of LDPE.
- Step 4 (t = 5 min): addition of 6 wt% of LDPE and 1.5 wt% of MA-g-PE.
- Step 5 (t = 9 min): end of the mixing process.

After compounding, the sorghum-PE blend were granulated in a Hellweg granulator with a 5-mm mesh and then kept in an air-circulating oven at 60°C until required to eliminate any moisture absorbed the composites during the process. Test bars were injection-molded in a Haake Minijet-II injection molding machine (Thermo Scientific, USA), with steel molds complying with either ISO-527-2-1BA (for tensile bars – dog-bone strips) or ISO-179 (for impact bars) specifications. Temperature was set at 150°C for the barrel and at 40°C for the molds. Composites were allowed to melt inside the barrel for 3 min before injection. Test specimens were injected under 600 bar pressure for 15 sec and maintained in the post-injection step at 40 bars for 10 sec. To avoid any deformations of the test bars, compressive air was used to assist the release from the mold.

2.4 Mechanical testing

Composite test bars were resting in a conditioned room at 23 ± 2°C and 50 ± 4 % relative humidity for at least five days before performing the mechanical tests. Tensile tests were carried out in a Zwick Z2.5 tensile testing machine (Zwick-Roell, Germany) with a force cell of 2.5 kN, operating at 0.02 mm/s (1.2 mm/min) with 55 mm gap between grips and 2 kN pre-tension. Young's modulus, ultimate tensile strength and elongation at break were measured. Charpy V-notch impact tests were performed with a pendulum Ceast 9050 (Instron, France) with a 1-J swinging arm. A 2-mm indent was made onto impact bars by a single tooth Ceast NotchVIS manual notching machine (Instron, France). Each reported mechanical result is the mean value of five tests.

2.5 Extraction of sorghum fragments after processing

Two composites, the one giving the best (genotype 9) and the one giving the worst (genotype 8) mechanical properties, were selected for the extraction of the sorghum fragments from the composites after processing. The LDPE matrix was dissolved in decahydronaphtalene (Decalin®) with the following conditions: about 1 g of the composite was added in 200 mL of Decalin® and then the mixture was heated up to 100°C and stirred at 400 rpm for approximately 1 h and 30 min. When the matrix was dissolved, the suspension was kept at rest and cooled down for at least 2 h before being used for size measurements. For the image acquisition, the obtained fragment/Decalin® suspension was diluted by adding more solvent in a proportion of 1:3 in order to decrease concentration, dissolve the remaining matrix and thus ensure the clarity of the suspension. The images were taken with an Epson Perfection™ V550 Photo Color Scanner (Epson, France), in transmission mode and with a resolution of 6400 dpi, corresponding to 4µm/pixel. The image analysis was performed using ImageJ software (a free Java image processing program, developed by National Institute of Health,

USA), 'Morphology' and 'Geodesics' plugins being used to measure fragment size. For each fragment, software provided the geodesic diameter *D*, the length *L* and the geodesic elongation, *L/D*. For each composite, about 12,000 fragments were measured for sizes between 4 and 50 µm and about 500 fragments for sizes above 50µm. Further information about the protocols used for sample preparation and image acquisition and treatment are given in (Di Giuseppe, 2016).

2.6 Biochemical analysis

The determination of cellulose and hemicellulose contents was based on the following protocols: 100 mg dry weight (DW) of ground samples (<100 µm) were washed twice in 5 mL of distilled water at 80°C. After centrifugation (10 minutes, 10.000 rpm), the pellets were rinsed twice in 5 ml of absolute ethanol for 15 min at 80°C, then rinsed twice in 5 mL of acetone at room temperature for 10 min and left to dry under a fume hood overnight at room temperature. The residual content was weighed to determine the percentage of Cell Wall Residue in the dry matter (CWR). For hemicelluloses hydrolysis, 500 µL of 2.5 M trifluoroacetic acid (TFA) were added into 10 mg of this CWR and the mixture was heated for 2 h at 100 °C. To determine the cellulose content, the residual pellet obtained after the TFA hydrolysis was rinsed once with 3 mL of distilled water, twice with 1.5 mL ethanol and the last rinse with 1.5 mL acetone, then left to dry under a fume hood overnight at room temperature. Cellulose hydrolysis was carried out with 250 µL of H_2SO_4 72% for 2h at ambient temperature, followed by the addition of 1.5 mL of distilled water for 2 h at 100 °C. Monosaccharides released by TFA and H_2SO_4 hydrolysis were diluted by 500 times and quantified using an HPAEC-PAD chromatography as described in (Harholt et al., 2006). The hemicellulose and cellulose contents were determined by the sum of their constituent sugars.

Glucose, fructose and sucrose contents were measured from 20 mg DW of ground samples. Powder was extracted with 1 mL of 80% ethanol for 30 min at 78°C, and then centrifuged (10 minutes, 10.000 rpm). The supernatant containing sugars was placed in 50 mL graduated flask. The pellet was suspended in 1 mL of 80% ethanol in same condition as previously, and this procedure was carried out three times. The flask containing the supernatant was adjusted with distilled water. After homogenization and filtration with membrane filter 0.22 µm, mono and disaccharide contents were measured using an HPAEC-PAD chromatography (Dionex, Salt Lake City, UT, USA). The separation was carried out by CarboPack PA1 column at 30°C with an isocratic elution of 150 mM sodium hydroxide.

The protocol for lignin determination was adapted from (Fukushima and Hatfield, 2001). Lignin from the prepared cell wall residue (5 mg +/- 1mg) was solubilized in 1mL of acetyl bromide solution (acetyl bromide/acetic acid (1/3, v/v)) in a glass vial at 55°C for 2.5 h under shaking. Samples were then let to cool down at room temperature and 1.2 mL of NaOH 2M/acetic acid (9/50 vv) was added in the vial. 100 µL of this sample was transferred in 300 µL of 0.5M hydroxylamine chlorhydrate and mixed with 1.4 mL of acetic acid. The absorbance (A280) of the samples was measured at 280 nm. Lignin content was calculated using the following formula:

$$\%lignin = \frac{100 \times (A280 \times V_{reaction} \times V_{dilution})}{20 \times V_{sample\ solution} \times m_{sample\ mg}}$$

All concentrations are provided in mg/g of dry matter with the exception of the Cell Wall Residue in the dry matter (CWR) which is provided in % of dry matter.

3 Results and discussion

3.1 Size distribution of the selected fractions

Since it has been observed that the diameter of plant fibers are affecting their mechanical properties (the larger the diameter is, the poorer the mechanical properties are (Sena Neto et al., 2015; Yan et al., 2014), it is important to control the sizes of fragments for all the genotypes studied. After being ground, the sorghum stem fragments were sieved and the size and aspect ratio of the fragments were measured by optical microscopy. Mean (or number average) and weight average were assessed for length and width values. Figure 1 shows the microscopy image of fragments passed through a 300 µm sieve and retained on the 200 µm sieve for one of ten genotypes. As can be seen on Figure 1, although sieving was applied, the length of the stem fragments varied due to the fact that sieving was selecting fragments on diameter, not on length. However, if a long fragment is mainly staying parallel to the sieve, it will not go through sieve holes despite its diameter is smaller than the sieve holes. All these factors were giving a rather large size distribution of fragments for each genotype despite the small size difference between two adjacent sieves. The aspect ratio of the stem fragments

(length over mean diameter) is affected by three factors: the histological properties and chemical composition of sorghum stems, the milling and the sieving processes. Sorghum stems were easy to break, producing a large amount of dust. The milling time was controlled and the two-step sieving process was ensuring the elimination or at least the minimization of dust or fine particles with no reinforcing capacity from the selected fraction. Figure 2 and Table 2 summarize the geometrical dimensions of the fragments obtained for one genotype. No significant differences in dimensions of stem fragments between genotypes were observed after passing through the milling/sieving processes. As seen in Figure 2, although there were differences in distribution of lengths, there was not a very large dispersion of aspect ratios, and aspect ratios from different sieves were very similar. Following a set of preliminary experiments with sorghum and other plants (maize and miscanthus), it was decided to use the 200-300 µm fraction to prepare composites with LDPE. As will be seen later, fragments collected on the 200 µm sieve provided low but measureable reinforcing capacity and composites had sufficient strengths to evaluate reinforcing differences (if any) between genotypes.

3.2 Biochemical composition of the selected fractions

Grinding produced fragments with a large dispersion of sizes. This comes first from the personal history of mechanical stresses encountered by each stem piece. Depending on the number of shocks and their magnitude and direction, pieces will break or not. Even if the starting material to be broken would be perfectly homogeneous, such grinding would produce pieces with a certain size and shape dispersion. In the case of sorghum stems, there is an additional factor, which is the high heterogeneity of stems themselves, with different tissues of different mechanical characteristics, distributed in a complex manner in the stem. Thus, the fact that these different tissues will break and disperse (i.e. reduce their size) in different modes should be expected. If it is true, some biochemical composition variations with fragment sizes should be detected. Figure 3 gives the variations of soluble sugars of the dry matter and of the different components of the cell walls, i.e. cellulose, hemicellulose, lignin for various fragment sizes for the three genotypes. The graphs on the right hand side of Figure 3 show the genotype effects detected for all the analyzed traits between the three varieties, which were expected since the genotypes were chosen to offer large composition differences. The situation regarding the effects of the fragment sizes is more contrasted. The graphs on the right hand side of Figure 3 show that for a given genotype, there was a rather strong variation of the different component compositions with fragment size. However, this variability was not statistically significant when all the genotypes are considered together (graphs on the left hand side of Figure 3). It means that fragments were breaking in a way that was selecting more or less some tissues, but that the different genotypes harbored different behaviors. The variation of lignin content was less pronounced from one fragment size to another for a given genotype with no statistical fragment size effect. The only detectable effect of sieving was on soluble sugars where the fragment size effect can be statistically detected. The general picture is that the genotype effect is larger than the sieving effect, which is a very important result regarding the possibility to assess genotype characteristics for the preparation of composites. The fact that no general trend of the sieving effect was detected can probably be attributed to the different behaviors of the three genotypes analyzed. It is interesting to remind here that one of the genotypes is a mutant which presents an impaired lignin biosynthesis.

3.3 Mixing process

Figure 4 is an example of the typical torque-time curves observed during the compounding process. The torque shows variations which correspond to the subsequent incorporation steps. Figure 4 shows two different compounding experiments, called LDPE+1 and LDPE+9, conducted with genotypes 1 (IS19453) and 9 (RE1), respectively. The torque sequence was very similar for these two examples, as it was for all compounds which were prepared, suggesting that the designed compound production gave highly reproducible results. Thermogravimetric results (not reported in this paper) showed that the first distinctive mass loss corresponding to the thermal degradation of soluble sugars started at 150-170°C, depending on the genotypes. The temperature measured in the mixing chamber, which is the dotted curve in Figure 4, shows that no significant thermal degradation of sorghum stem fragments was expected since temperature was always below 160°C.

3.4 Mechanical properties

Figure 5 is showing typical stress/strain curves for composites obtained from two different genotypes and LDPE. The sorghum stem fragments have a reinforcing effect leading to an increase in tensile strength and modulus. On the other hand, this increase in modulus and strength is at the expense of a very large reduction

of the elongation at break, as can be seen in Figure 5. The difficulty for properly interpreting mechanical testing is due to the fact that such composites cannot be well characterized in several aspects. The mechanical properties of the stem fragments are not the same from one fragment to another and are even varying from place to place inside each fragment due to intrinsic variations of their composition and structure (see Figure 3). In addition, their dimensions are not well defined (the "diameter" for example is not easy to characterize when an axial ratio L/D is calculated, as can be seen in Figure 1). Another difficulty, present in all composites but exacerbated here, is the ill-defined filler orientation and distribution after processing. For composites with low aspect ratio fillers, as it is the case for sorghum (Table 3), any small decrease of L/D is affecting the final strength of composites (Nystrom et al., 2007). Although some researchers tried to model or predict the mechanical performances of plant filled composites with results that compared reasonably well to experimental results (Andersons et al., 2006; Facca et al., 2007), all these models and most of the literature dealing with the relations of the plant filler with the mechanical properties of composites, have been using only concentration and aspect ratio of the fillers as the only parameters (Ku et al., 2011). Very few studies were effectively considering the effect of the microstructure of the filler on the properties of the composites. To our knowledge, it was only considered once in (Oksman et al., 2009) with a result stating that the microstructure of the fibers does not seem to influence the strength of composites. This is a rather counterintuitive result which may be due to the fact that authors studied different natural fillers without deeply investigating the inner structure of these fillers. The advantage of the present study is that filler concentration and filler size from one plant species were fixed at the beginning of the work, in order to concentrate on the variability of the biochemical composition of the filler. For the same reason, the interfacial properties were fixed (see below for a deeper discussion of this point) by using the same compatibilization agent, owing to the importance of this parameter on composite properties (Kabir et al., 2012; Kaewkuk et al., 2013). So any changes which would occur in the mechanical properties of composites were expected to be only related to the filler structure and chemical composition.

Several factors will contribute to variations of mechanical properties of the composites when using different genotypes. One is obviously the intrinsic mechanical resistance of the stem fragments (the whole stem properties is also depending on other parameters than fragment size distribution or aspect ratios), which may vary depending on composition and internal histological structure of the whole stem. This will directly impact the mechanical performances of the composite. Another linked phenomenon is the attrition of fragments when going through the small slits of the mixer and the injection molding tools. This reduction of size and potentially of aspect ratio, plus the possible production of very small fragments (dust-type product) will also have an effect. All this may also change the way these fragments are positioned in the test bar, creating or not a skin-core effect and varying the degree of orientation, known to be important factors (Ho et al., 2012). Finally, one cannot rule out the fact that the way stems are breaking might lead to various exposed tissues with different surface properties, changing thus the compatibilization. These are the questions which should be addressed when one tries to fully evaluate if there is a genotype effect on the mechanical performances of composites.
Figure 5 shows that the stress-strain curve of the composite has no plastic plateau, contrary to the case of the matrix polymer. Such pure elastic behavior is seen on most natural fiber-filled composites, which are very fragile (Yan et al., 2014). A similar result was obtained at high filler contents (above 20-30%) for hemp-polyethylene (Lu and Oza, 2013), for high density polyethylene composites reinforced with oil palm fibers (Kakou et al., 2014) and for alfa fiber-polypropylene (Arrakhiz et al., 2012). Figure 5 suggests that it is the filler which is controlling the mechanical behavior, bringing modulus and strength, but drastically reducing ductility. This is the common behavior of all plant fiber-filled composites. This figure also shows that some genotypes seem to be better than the others, which can be better seen in Figures 6 and 7. The mechanical properties of the different composites prepared are depicted in Figures 6 and 7 for tensile strength, Young's modulus, elongation at break and impact strength. The results show a large variation in the reinforcing capacity of the tested genotypes (coefficients of variation between 10.7 and 16.8 % depending on the trait). Two sorghum genotypes (3, IS26731 and 8, EUG341F: ES-ATHENA) show no reinforcing capacity, as their ultimate tensile strengths remain the same as that of neat LDPE matrix. Conversely, two other genotypes (4, IS30405 and 9, RE1) have a good strengthening capacity, with up to 60 % increases in ultimate tensile strength of composites compared to LDPE. The remaining genotypes present intermediate values with enhancements of 20-40 %. The variability of the measurements between the ten genotypes analyzed is given

in Table 3. The Young's modulus, tensile strength and elongation at break show similar coefficients of variation (15.8-16.8 %) whereas the coefficient of variation observed for impact strength is lower (10.7 %). This suggests that the impact strength could be controlled by physical parameters different from the ones for the Young's modulus, tensile strength and elongation at break.
Since there is a linear strength-stiffness relationship (Figure 6), as it is commonly seen with short fiber filled composites including bio-based ones (Sobczak et al., 2012), the tendencies observed in the analysis of the ultimate tensile strength are also seen for the Young's modulus. Thus, as could be thought, the genotypes with higher reinforcing capacity are the ones with higher rigidity and higher modulus. Genotypes 3 (IS26731) and 8 (EUG341F: ES-ATHENA) present the lower values of elastic modulus, tensile strength and impact strength, while the higher values of Young's modulus and strength are for genotypes 9 (RE1) and 4 (IS30405). This ranking is different for the impact strength for which the highest performances are for genotypes 6 (BN612) and 5 (IS30417), suggesting again that different tissues, histological organization and/or biochemical compositions are involved in the tensile and impact resistances. Given the extremely low rigidity of LDPE, increases of over 400 % can be seen even by the sorghum genotype with the worst reinforcing capacity. What is observed here with the sorghum stem composites is following the general picture of the mechanical properties of plant filler-polyethylene composites. With few exceptions, adding a plant based filler like pine, sugarcane bagasse, rice husk and straw (Liu et al., 2013), cellulose pulp (Sdrobiş et al., 2012), hemp (Facca et al., 2007; Lu and Oza, 2013), sisal (Zhao et al., 2014), alfa (Arrakhiz et al., 2012), coir (Zhao et al., 2014), date palm wood powder (AlMaadeed et al., 2014), coconut (Brahmakumar et al., 2005), doum (Arrakhiz et al., 2013) and rayon (Ganster et zl., 2006) is increasing Young's modulus and sometimes tensile stress and decreasing strain at break and impact resistance. The mechanical properties are strongly influenced by the properties of the filler as soon as the adhesion between the filler and the matrix is strong.
Table 4 gives the variations of modulus and strength and the values of elongation at break and notched impact strength for various plant species filling polyethylene when the filler concentration is at 20-30 %. As can be seen, the improvement of modulus due to sorghum is in the range of what can be seen for other plant-based fillers. The strength is not improved, as for most of the other species fillers, with the exceptions of rayon and sisal which improved the composite properties by a minimum magnitude of two. Sisal has an intrinsic tensile strength between 500 and 600 MPa (Faruk et al., 2012) and the one of rayon used in (Ganster et al., 2006) is 825 MPa, compared to coir 175-220 MPa (Faruk et al., 2012; Satyanarayana et al., 2009), and bagasse 20-290 MPa (Jawaid and Abdul Khalil, 2011). Table 5 suggests that the sorghum stem fragments have a low strength. The elongation at break of sorghum composites is rather large, due to the low strength of composites. One of the major difficulty in using plant-based composites is the low impact strength, in the order of a few kJ/m^2 for most composites including the ones prepare with sorghum, with the notable exception of the man-made fiber rayon-based composites which reach 22 kJ/m^2.

Tensile strength and Young's modulus

Tensile strength and Young's modulus are showing a dependence on genotypes. As observed in Figure 4, the mixing torque changed depending on sorghum genotype during the blending/compounding process, although it was with the same amount of materials, adding sequences, and processing conditions. The addition of sorghum stem fragments from genotype 9 (RE1) created higher torque than the addition of those from genotype 1 (IS19453). Therefore, the power consumption during mixing should also vary with sorghum genotype. This is indeed the case and Figure 8 shows clearly that the higher the Young's modulus and the strength are, the higher the power consumption during mixing is. One way to interpret these results would be to consider the viscosity of the mix since the torque is directly linked to it. It seems that the different genotypes exhibit a variability at this step leading to a large variation of the breakage extend between them. At the beginning of the mixing, all genotypes have about the same size distribution. During mixing, stems fragments are broken in smaller pieces, and the extend of breakage varies with genotypes. In other words, some stem fragments are stronger than others. When being broken, there are more fragments with smaller length, and this decreases the viscosity due to the decrease of inter-stem fragment topological contacts during flow. Since strength and modulus depend first on the intrinsic properties of the filler and second on their aspect ratio, the weakest fragments would see a decrease of aspect ratio and have lower intrinsic mechanical properties, these two factors decreasing the mechanical properties of the composites. This is confirmed by the fact that there is a direct correlation between the energy consumption during mixing and the mechanical properties of the composites. Figure 8 shows that it is more difficult to mix blends which will give the best

mechanical properties after processing. It is indeed what is observed when the dimensions of the stem fragments after processing for genotypes 8 (EUG341F: ES-ATHENA) and 9 (RE1) were measured. Table 5 shows that the composites made with genotype 9 (RE1) has fragment dimensions after processing larger than the composite made with genotype 8 (EUG341F: ES-ATHENA).

4 Conclusions

Sorghum stem fragment-polyethylene composites can be easily prepared. Ten genotypes having different origins and biochemical compositions were used and they could be compared owing to a robust preparation protocol. They have mechanical properties in line with what has been reported in literature for biomass-based polyethylene composites. A variation of biochemical properties with fragment sizes was evidenced. Starting from the same fragment size, composites showed a variation of mechanical properties depending on genotypes. This was mainly due to the fact that fragments were broken during processing and that some genotypes showed more resistance to breakage than the others. The genotypes having the highest viscosity during mixing are the ones having the highest tensile mechanical properties and the less decrease of size after processing. This ranking did not seem to fully apply for impact resistance, suggesting that different tissues, histological organization or biochemical compositions are involved in the tensile and impact resistances.

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Figure Captions

Figure 1. Microscopy images of sorghum stem fragments collected at 300 and 200 µm sieves.
Figure 2. Size distribution of sorghum stem fragments collected at three different sieves (between 100 and 200 µm, 200 and 300 µm and 300 and 400 µm) in terms of length (left hand side graph) and aspect ratio (right hand side graph) for one sorghum genotype (BIOMASS140, genotype 7).
Figure 3. Effects of sieving (left hand side graphs) and genotype (right hand size graphs) on the components of stem fragments for three genotypes. A: cellulose. B: hemicellulose. C: lignin. D: soluble sugars
Figure 4. Torque-temperature-time curves during compounding of sorghum stems with LDPE. LDPE+1 and LDPE + 9 are two different compounding experiments with genotypes 1 (IS19453) and 9 (RE1).
Figure 5. Stress/strain curves of the LDPE and two composites produced with genotypes 3 (IS26731) and 9 (RE1).
Figure 6. Tensile strength and Young's modulus of the LDPE-sorghum reinforced composites (standard errors correspond to five test bar replicates per genotype) (A) and the relationship between the two parameters (B).
Figure 7. Elongation at break (elongation at break of LDPE was not detected) (A) and notched impact strength of sorghum-LDPE reinforced composites (notched impact strength of LDPE = 44.7 ± 1.1 (kJ/m^2)) (B). Standard errors correspond to five test bar replicates per genotype.
Figure 8. Correlations between energy consumed during compounding and tensile strength of composites (A) and energy consumed during compounding and Young's modulus (B) of the composites, for the ten genotypes.

Table 1. Characteristics of the ten genotypes

Genotype-partner-code	Code in this article	Seed origin	Race	Country of origin	Genotype type	Phenotypic characteristics (based on previous phenotyping trials)
IS19453	1	ICRISAT	Durra	Bostwana	Pure line	High NDF digestibility, low lignin content, high tolerance to pre- and post-flowering drought stress
IS20351	2	ICRISAT	Durra	Nigeria	Pure line	High juice content, high lignin content, low NDF digestibility
IS26731	3	ICRISAT	Bicolor	South Africa	Pure line	High sugar juice content, relatively low NDF content
IS30405	4	ICRISAT	Caudatum-bicolor	China	Pure line	Low juice content, high NDF content with low NDF digestibility
IS30417	5	ICRISAT	Caudatum-bicolor	China	Pure line	High lignin content
BN612	6	Commercial line	Caudatum	NA	Pure line	Double bmr mutant Bmr6 + Bmr12: low lignin content and high NDF digestibility
BIOMASS140	7	Eurosorgho	mixed	NA	Commercial hybrid: industrial use	High biomass production, high cellulose and sugar content
EUG341F : ES-Athena	8	Eurosorgho	mixed	NA	Commercial hybrid: silage use	High biomass production with high *in vitro* matter digestibility
RE1	9	Eurosorgho	NA	NA	Male parent of BIOMASS140	High NDF, low organic matter solubility, low NDF digestibility
AE1	10	Eurosorgho	NA	NA	Female parent of BIOMASS140	Short stem, high grain production, high digestibility biomass and NDF digestibility

(NA: Not available, NDF: neutral detergent fiber)

Table 2. Dimensions (*L*, *D*, *L/D*) of selected sorghum stem fragments after milling and sieving (BIOMASS140, genotype 7). Indices *n* and *w* designate the mean and the weighted average values, respectively.

Lower Sieve Mesh size	L_n (µm)	L_w (µm)	D_n (µm)	D_w (µm)	$(L/D)_n$ (-)
100 µm	590	740	120	130	5.5
200 µm	900	1210	190	207	5.5
300 µm	1430	1940	240	260	7.0

Table 3. Variability of Young's modulus, tensile strength, impact strength and elongation at break of composites prepared with the ten genotypes

	Young's modulus (GPa)	Tensile strength (MPa)	Impact strength (kJ/m^2)	Elongation at break (%)
Mean value	0.75	9.5	5.5	36.7
Standard deviation	0.12	1.5	0.58	6.2
Coefficient of variation (%)	16.0	15.8	10.7	16.8

Table 4. Ratio of the Young's modulus of the composite over the Young's modulus of the used PE, ratio of the strength of the composite over the strength of the used PE, elongation at break and impact strength for various composites having a PE matrix and different plant species based fillers

Plant type (% in composite)	Modulus comp/modulus PE (-)	Strength comp/strength PE (-)	Elongation at break (%)	Impact strength (kJ/m^2)	Reference
sorghum 30 %	4.5-7.5	1-1.6	~30	5-6	This work
rayon 25 %	3	3.5	--	22	(Ganster et al., 2006)
doum 30 %	2.5	0.8	40	--	(Arrakhiz et al., 2013)
sisal 30 %	3-4	3-4	4	--	(Zhao et al., 2014)
coir 20 %	1.5-2	1-1.4	--	--	(Arrakhiz et al., 2012)
pine flour 30 %	3.6	1.3	--	4.7	(Liu et al., 2013)
bagasse 30 %	5.5	1.5	--	5	(Liu et al., 2013)
rice husk 30 %	4.6	1.3	--	4	(Liu et al., 2013)
rice straw 30 %	3.5	1.2	--	4.5	(Liu et al., 2013)

Table 5. Mean and weighted average lengths (L_n and L_w), diameters (D_n and D_w), aspect ratios (($L/D)_n$ and $(L/D)_w$) of fragments extracted after processing from two composites prepared with genotypes 8 (EUG341F: ES-ATHENA) and 9 (RE1). Two classes were selected to measure dimensions, fragments with a size above 4 µm and fragments above 50 µm. The Young's modulus, the tensile strength and the notched impact strength of composites prepared with the two genotypes are also given.

	L_n (µm)	L_w (µm)	D_n (µm)	D_w (µm)	$(L/D)_n$ (-)	$(L/D)_w$ (-)	Young's modulus (GPa)	Tensile strength (MPa)	Impact strength (kJ/m^2)
PE+8							0.6	7.2	4.9
≥ 4 µm	82	318	35	66	2.1	3.5			
≥ 50 µm	140	380	52	83	2.7	4.6			
PE+9							1	11.7	5.8
≥ 4 µm	100	460	35	76	2.3	3.6			
≥ 50 µm	200	4540	57	100	3.2	4.8			

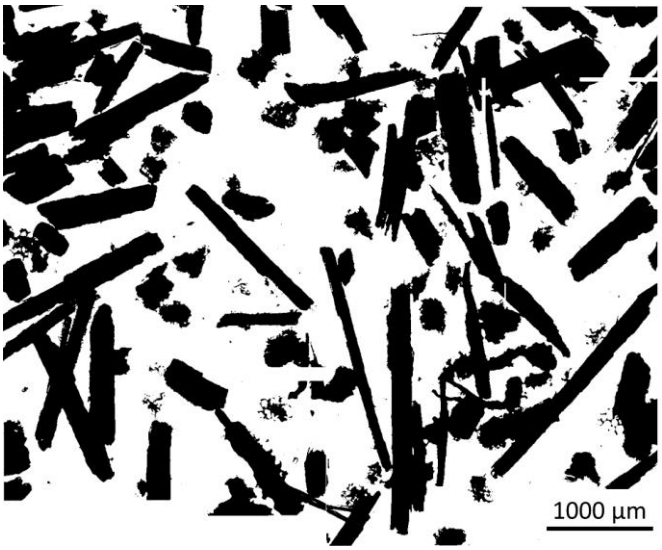


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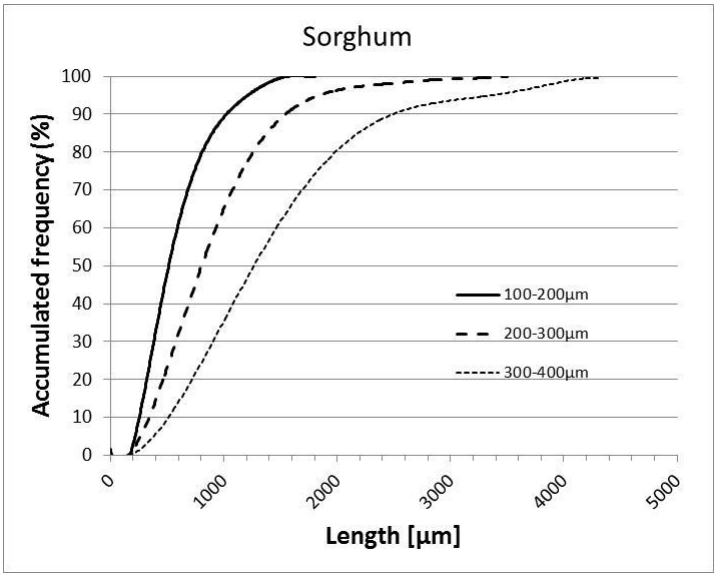


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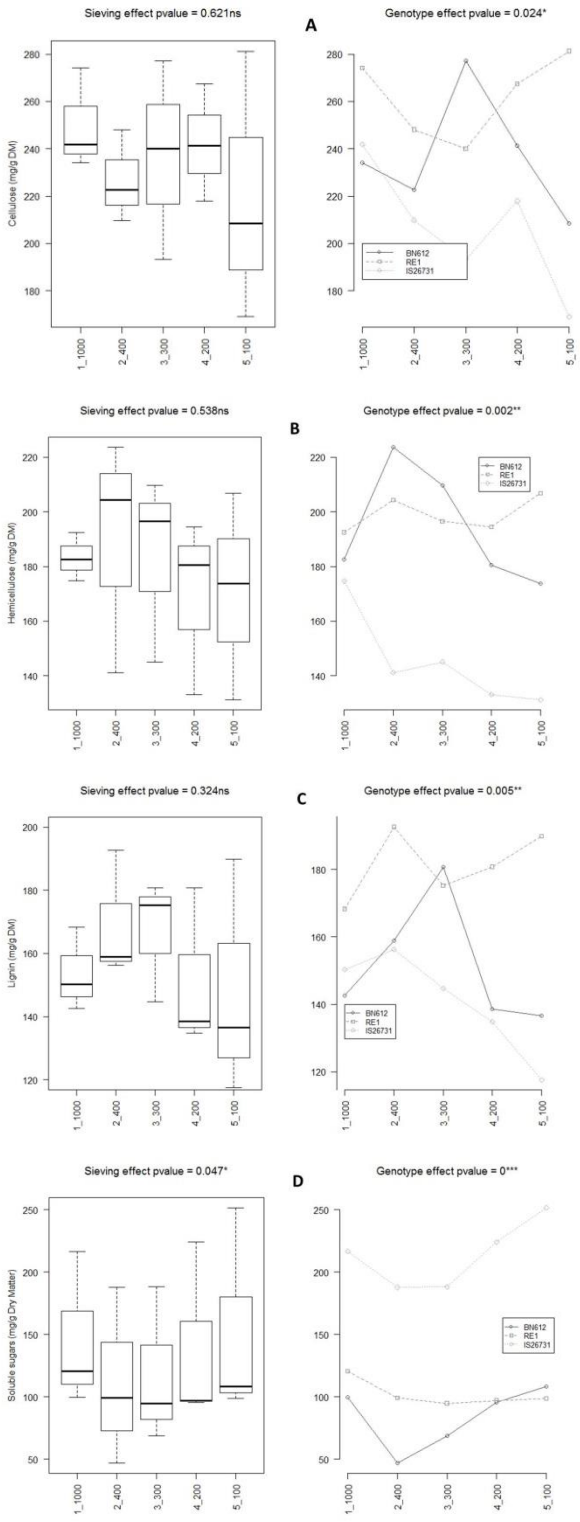


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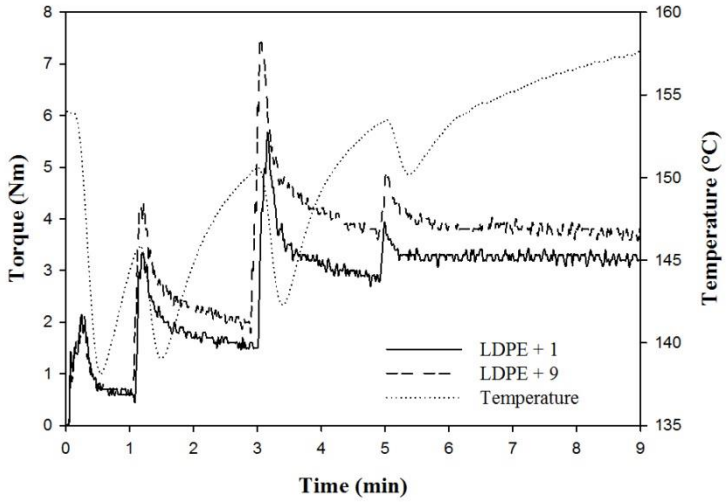


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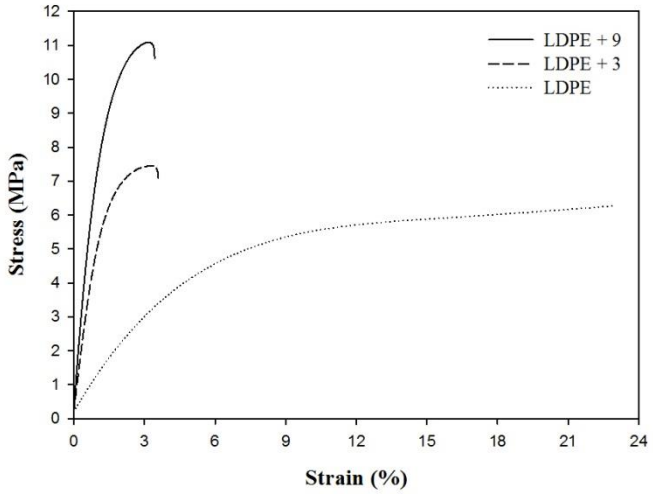


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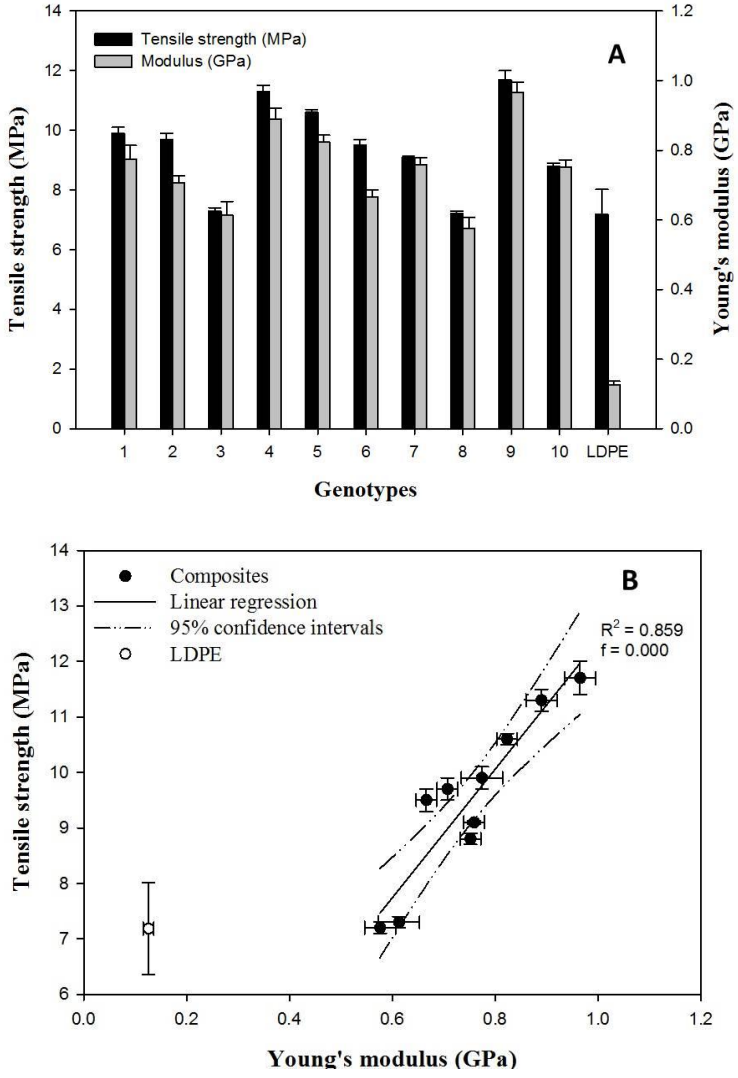
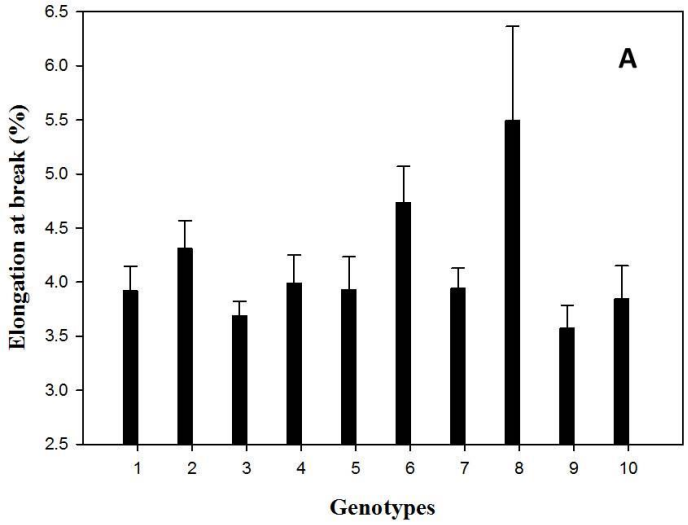


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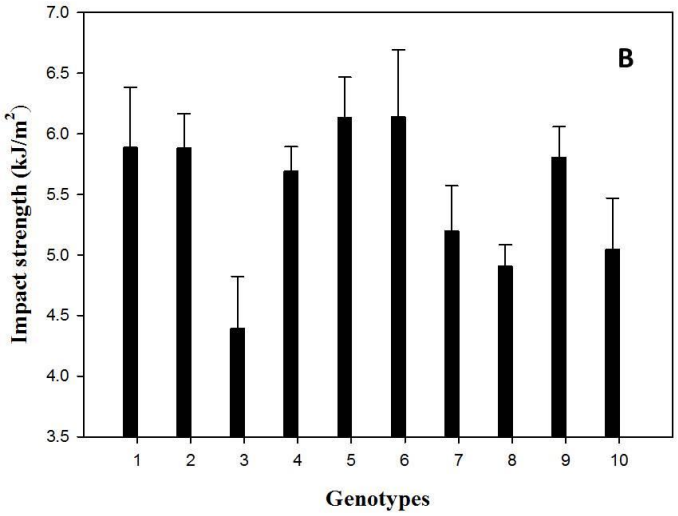


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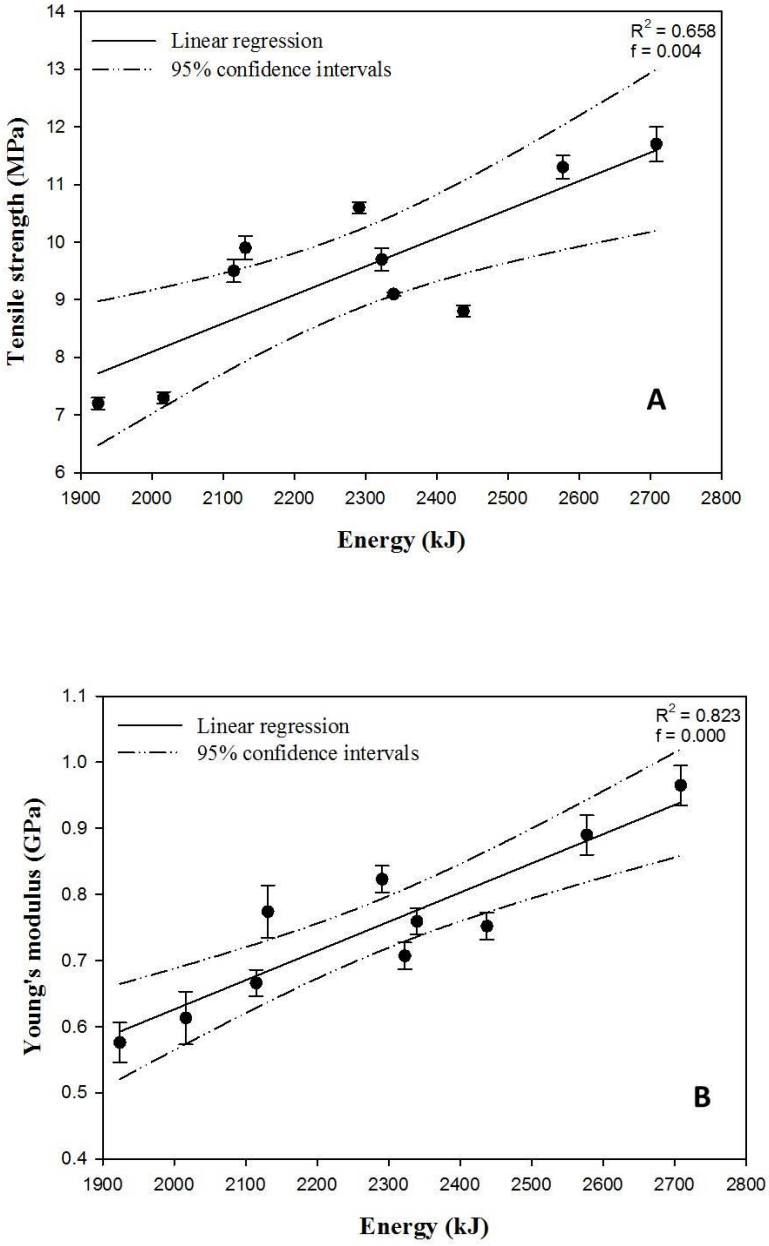


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