The Influence of Fines Material on the Mechanical Performance of Wood Fiber Polypropylene Composites

David Sandquist,[a,*] Armin Thumm,[b] and Alan R. Dickson [b]

With an increasing interest in a wider scope of biomass feedstocks for biocomposite production, the potential influence of a possibly higher proportion of fines material needs to be addressed. In the pulp and paper industry, at least two major classes of fines material are identified; blocky fines and fibrillar fines. These differ in their aspect ratio, with the later having substantially higher aspect ratio. In this study, the physical impact of blocky fines (from CTMP refining) and fibrillar fines (produced via Super® Masscolloider refining) on a wet formed compression molded polypropylene (PP) matrix biocomposites were compared. The results indicated that in wet formed compression molded polymer matrix composites, both blocky (CTMP) and fibrillar (SMC) fines have a significant negative impact on modulus of rupture (MoR). Additionally, blocky CTMP fines also have a significant negative impact on modulus of elasticity (MoE). It is postulated that this is due to fibril agglomeration, in the case of fibrillar fines, and low aspect ratio in combination with some agglomeration, in the case of blocky CTMP fines. The indication is that fines material that has not been treated to minimize agglomeration has limited benefits as a reinforcing agent, and only a negative impact on most properties in wet formed compression molded polymer matrix composites.

Keywords: Composites; Biocomposites; Polymer-matrix composites; Pulp fines; Fines material

*Contact information: a: VTT Technical Research Centre of Finland Ltd, P.O. Box 1000, FI-02044 VTT, Espoo, Finland; b: Scion, Te Papa Tipu Innovation Park, 49 Sala Street, Rotorua 3046, New Zealand; * Corresponding author: david.sandquist@vtt.fi*

INTRODUCTION

Scientific interest and commercial markets for biocomposites have grown significantly over the last two decades, and the market is projected to double in volume over the next 5-year period (Carus *et al*. 2015; Harlin *et al*. 2018). The interest in biocomposites is driven by resource scarcity, environmental awareness, reduced CO_2 footprint, and recycling incentives (predominantly in the EU) (Harlin *et al*. 2018). The first-generation biocomposites were exemplified by wood flour / polyethylene or polypropylene matrix wood plastic composite (WPC) decking. Wood flour provides increased composite stiffness without significant improvements in other physical or mechanical properties due to the low aspect ratio of the particles (Chen *et al*. 2006; Nygård *et al*. 2008; Peltola *et al*. 2014). To achieve strength reinforcement and increased impact resistance, a fiber with a higher aspect ratio is required, such as wood pulp fibers (Nygård *et al*. 2008; Peltola *et al*. 2014).

In pulping, a cellulosic fraction of smaller fiber fragments (called fines) is commonly produced (Biermann 1996), and is defined as the fraction that is able to pass through a 76 μm opening of a Britt Dynamic Drainage Jar (Kari *et al*. 2016). The amount and morphology of fines material varies greatly between different pulping and refining

processes (Odabas *et al*. 2016).

Fines play a central role in physical paper properties, particularly for mechanical pulp based paper (Odabas *et al*. 2016). The effect of the fines particles is analogous to putty, bridging the fiber-fiber joints (Ek *et al*. 2009). Fines are known to aggregate strongly when processed under aqueous conditions (Ek *et al*. 2009). However, in composites the reinforcing fibers should ideally be well dispersed in the matrix, for optimal reinforcement potential (Peltola *et al*. 2014).

Fines materials are situated on a spectrum between wood flour and nanocellulose in size. Wood flour are ground wood particles in the range of approximately 99 to 795 µm in diameter (Lee *et al*. 2014), whereas micro- or nanofibrillated cellulose commonly have a width of 10 to 100 nm and length of 0.5 to 50 µm for the former, and width of 4 to 20 nm and length 0.5 to 2 µm for the latter (Sandquist 2013).

In the case of wood flour, the particle size distribution (and wood species) has a significant influence on the physical properties of a wood flour / polymer matrix composite (Stark and Berger 1997a,b; Stark and Rowlands 2003). Wood flour content up to 40% by weight yielded an increase in physical properties, after which properties started to plateau (Stark and Berger 1997a). Stark and Berger reported that smaller diameter wood flour particles gave an higher increase in strength, elongation, and stiffness compared to larger particles (Stark and Berger 1997b), especially comparing 60 µm with 500 µm fractions.

Similarly, the reinforcement potential of nanocellulose in polymer matrix composites depends greatly on the particle size distribution and aspect ratio (Sandquist 2013; Lee *et al*. 2014). Bacterial-, micro-, and nano-fibrillated cellulose are particles with very high aspect ratios (Sandquist 2013). In addition, all native nanocellulose materials are susceptible to irreversible agglomeration if dried from an aqueous suspension. For nanocelluloses to be able to increase physical or mechanical performance in a polymer matrix composite, it is vital that they are well dispersed (Islam *et al*. 2013). There is a linear relationship between nanocellulose addition and physical reinforcement of bacterial and nanofibrillated cellulose composites, following a rule-of-mixtures relationship (Madsen *et al*. 2009; Lee *et al*. 2014) with both types of materials achieving significant reinforcements.

While the importance and impact of fines in pulp and paper production is well known (Ferreira *et al*. 1999; Lin *et al*. 2007; Hafrén *et al*. 2013; Hyll 2015; Mayr *et al*. 2017), few studies have addressed the impact of fines in polymer matrix composites.

Fekete *et al*. (2018) observed substantial physical reinforcement with low aspect ratio cellulose fibers in thermoplastic starch, as did Toriz *et al*. (2002) with lignin particles in polypropylene (PP). However, both Peltola *et al*. (2011, 2014) in PP and polylactide (PLA) and Gallagher and McDonald (2013) in polyethylene (PE) showed negative effects on the physical reinforcement of composites with fines or very small wood particles.

In modeling of polymer matrix wood fiber composites, fines are often disregarded (Miettinen *et al*. 2012, 2015; Newman *et al*. 2014; Miettinen 2016), as they are considered to have a limited or no contribution to reinforcement of strength or stiffness of the composite (Miettinen 2016).

With more fines material being incorporated in biocomposites, it is increasingly important to establish what their contribution is. This study examined the influence of two structurally different fines materials, blocky and fibrillar, on a wet formed compression molded PP polymer matrix composite. Wet formed compression molding was selected as an alternative to dry compounding, as wet forming allowed blending of the fines with PP fibers without prior drying, in an effort to minimize any agglomeration caused prior to the composite formation.

EXPERIMENTAL

Materials

Fibers and fines

The blocky fines used in this study were collected from a (*Pinus radiata* D. Don) commercial chemi-thermal mechanical pulp (CTMP) supplied by Winstone Pulp International (Ohakune, New Zealand) in a dry form. The pulp was prepared by a standard pulp disintegration (AS/NZS 1301.203s:2007 (2007)) then separated into long fiber and fines fractions using a device compliant with TAPPI T 261 (1994) fitted with a 100-mesh screen. For each run, 100 g (oven dried basis) was washed through the screen in 10 L of water. The fines were allowed to settle for several hours before excess water was decanted.

The fibrillar fines were produced from a (*P. radiata*) bleached kraft pulp (OJI Fiber Solutions, Kinleith, New Zealand) by passing it through a Super® Masscolloider (SMC) (Masuko Sangyo, Co., Ltd, Kawaguchi, Japan) 47 times at 4% consistency (Kang and Paulapuro 2006). The CTMP fiber and two different fines material were combined at the percentages shown in Table 1.

Table 1. Fiber and Fines Blends

CTMP long fiber (%)	CTMP fines (%)	SMC fines (%)
100	0	0
71	29	0
50	50	0
25	75	0
75	0	25
50	0	50
25	0	75

Polymer

Polypropylene (PP) fibers (Atofina) were provided by FiberVisions (Covington, Georgia, USA). The PP fibers were 23 µm wide and 5 mm long with a melting point (by DSC) of 165 to 166 °C. The fibers include an undisclosed coupling agent.

Methods

Compression molding of composites

Polypropylene (PP) fibers and the appropriate fiber/fines combination were weighed to give a 60% PP loading (wt%). The PP fibers and the fiber/fines were rapidly stirred with a pulp disintegrator (British Pulp Evaluation Apparatus, Mavis Engineering Ltd., London, UK) separately in water. The rapid stirring was performed for 10 min each. The PP and fiber/fines were then combined using an overhead stirrer for several minutes. The combined fibers were formed into 17 cm square pads using a forming box. After evacuation of the water the pads were air dried for storage and then dried overnight at 60 °C before compression molding. Pads were compression molded at 180 °C for 3 min using a 3 mm spacer to define the thickness of the panel. A force of 200 kN (~7 MPa) was sufficient. Three panels were made for each treatment. From each panel six flexural test samples (13 × 130 mm) were cut.

Fiber and fines analysis

Fiber lengths and widths were measured using a FiberLab 3 (Neles Field Controls Inc, Kajaani, Finland). Fines material was stained with safranin (BDH, England) (approx. 0.1%) and mounted on glass slides using Eukitt resin (O. Kindler, GmbH, Freiburg, Germany). Differential interference contrast illumination was used to highlight the fibrillar material on a Leica DMRB microscope fitted with a Leica EC3 digital camera (Leica Microsystems, Singapore). Dimensions were measured using ImageJ 1.51k (Schindelin *et al*. 2012; Schneider *et al*. 2012).

Mechanical characterization of composites

Densities of samples were determined using a water displacement technique according to ASTM D 792 (2008). Flexural properties were measured using an Instron 5566 testing machine (Instron, Norwood, MA, USA) according to ASTM D 790 (2003). The crosshead speed was 1.3 mm/min. The support span to depth ratio (L/d) was 16/1 giving a span of ~50 mm. Specimens were tested to failure to obtain the flexural modulus (Modulus of Elasticity (MoE)) and the flexural strength (Modulus of Rupture (MoR)). The values were normalized against density to correct for minor variations.

Microscopical imaging of composites

Representative cross-sections of the CTMP and SMC fines composites were prepared by grinding and polishing the composite using a range of grinding papers (320 to 4000 grit). The unstained composites materials were imaged using a Leica SP5 II confocal laser scanning microscope (Leica Microsystems, Mannheim, Germany). The excitation wavelength was 488 nm and emitted light was recorded between 500 and 598 nm. As PP has no autofluorescence, only the fibers are visible in the images, while the PP matrix remains dark. Two images were captured for each composite (2048 × 2048 pixels, ~390 × 390 µm). To measure agglomeration, the 8-bit greyscale images were segmented into a grid of non-overlapping squares with widths ranging from 40 to 200 µm. This equates to 100 squares per image (~40,000 pixels) for the smallest squares and 4 squares (~1,000,000 pixels) for the largest. The mean greyscale of the square was recorded. The standard deviation (std. dev.) of the mean greyscale values within a square indicates the variability across the image at the scale of the area of the square.

RESULTS AND DISCUSSION

Fines Characterization

The CMTP fines were a mixture of high aspect ratio fibrils and low aspect ratio fiber fragments (Fig. 1A). The SMC fines largely lacked the low aspect ratio particles and consisted largely of very fine, high aspect ratio fibrils (Fig. 1B). Due to the fineness and agglomeration of the SMC fines material, definite dimensions were not obtained; however, the mean aspect ratio of the visible fibrils was estimated to be > 55. This is much greater than aspect ratios measured for the CTMP fines (Table 2).

The particle sizes for fines fall in a continuum between wood flour (Liu *et al*. 2014) and micro- or nanofibrillated cellulose (Sandquist 2013). Nanocellulose has been well established to have a high reinforcement effect in paper made from wood fiber (Kajanto and Kosonen 2012; Brodin *et al*. 2014) and polymer matrix composites (Lee *et al*. 2014). Much less is known about fines in polymer matrix composites.

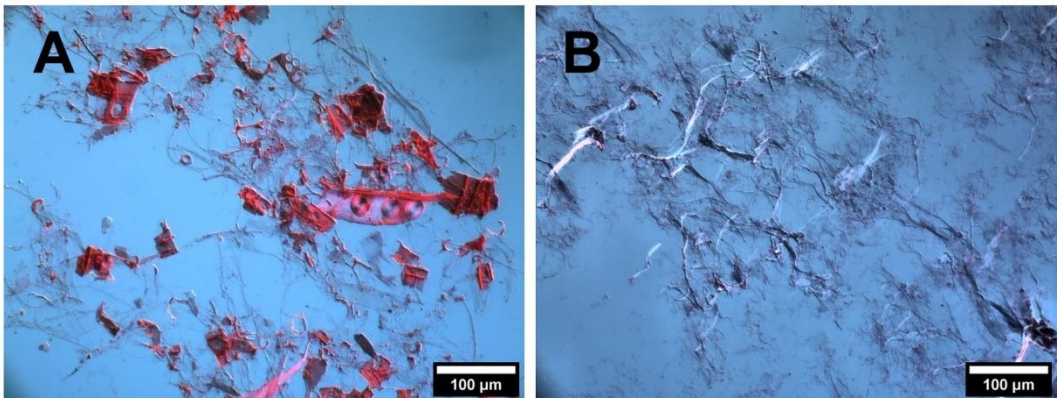

Fig. 1. A) Blocky chemi-thermal mechanical pulp (CMTP) fines and B) fibrillar Super Masscolloider cellulose (SMC) fines. Both are predominantly from a *Pinus radiata* source, with blocky fines collected from a CTMP pulp, and the fibrillar produced from a kraft pulp.

Table 2. Fiber and Fines Dimensions

	CTMP Fiber	CTMP Fines	SMC Fines
total number	10148	682	-
mean length (mm)	0.8725	0.0435	Est. >0.25
mean width (mm)	0.0293	0.0047	Est. <0.0045
mean aspect ratio	30.39	14.91	Est. >55
Length weighted length (mm)	1.3338	0.0001	-

The critical fiber length for *P. radiata* wood fibers has been estimated to be 0.8 mm (Thumm and Dickson 2013). This is equal to an approximate aspect ratio of (0.8/0.03) = 26. For simplicity it was assumed that the lignocellulose material in wood pulp, wood flour, fines, and nanocellulose display similar tensile strengths and interfacial shear strengths. Under this assumption, an aspect ratio of > 25 should give the maximum reinforcement in a polymer matrix composite. This means SMC fines display an aspect ratio that could achieve maximum reinforcement, whereas the CTMP fines do not (Table 2). Similarly, most micro- and nanocellulose materials show aspect ratios that can achieve maximum reinforcement (Sandquist 2013), whereas wood flour does not (Stark and Berger 1997b).

Homogeneity of the Wet-formed Compression Molded Composites

Wet-formed compression molding was selected as an alternative to dry melt polymer extrusion compounding to overcome the strong tendency of the fines material to agglomerate (Lin *et al*. 2007). If the fines had been dried prior to blending with the PP, it would have limited the analysis of the effect of dispersed fines.

While every effort was made to disperse the fines material homogeneously during the composite formation, it should be noted that it is difficult to eliminate minor losses or aggregation (or layering) of fines particles, particularly at high fines content. To estimate the integrity and homogeneity of the produced compression molded composite samples, density measurements and confocal microscopy were utilized.

The resulting mean density of all the materials showed that all fines/fiber compression molded composites samples were in the range of 1000 to 1022 kgm^{-3}, with no significant deviations (Fig. 2).

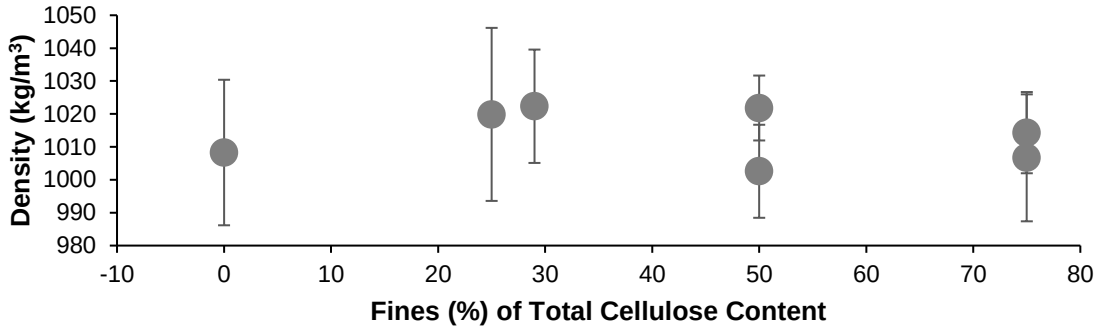

Fig. 2. Resulting densities of the compression molded composites show that there are no significant deviations in density profiles. Total number or samples were 7, with 3 replicates per data point.

Microscopic examination of representative cross-sections of the composites showed an overall higher level of agglomeration for SMC fines compared to the CTMP fines (Fig. 3). This can also be seen in the analysis of the greyscale values (Fig. 4). The CTMP fines composite had a lower standard deviation over all observed scales compared to the SMC fines composite. Thus, the cellulosic material in the CTMP composite was more evenly distributed than in the SMC composite. This effect was attributed to the higher aspect ratio, more fibrillar nature, and greater surface area of the SMC composite, resulting in greater bonding and agglomeration. No layering of the cellulosic materials was observed.

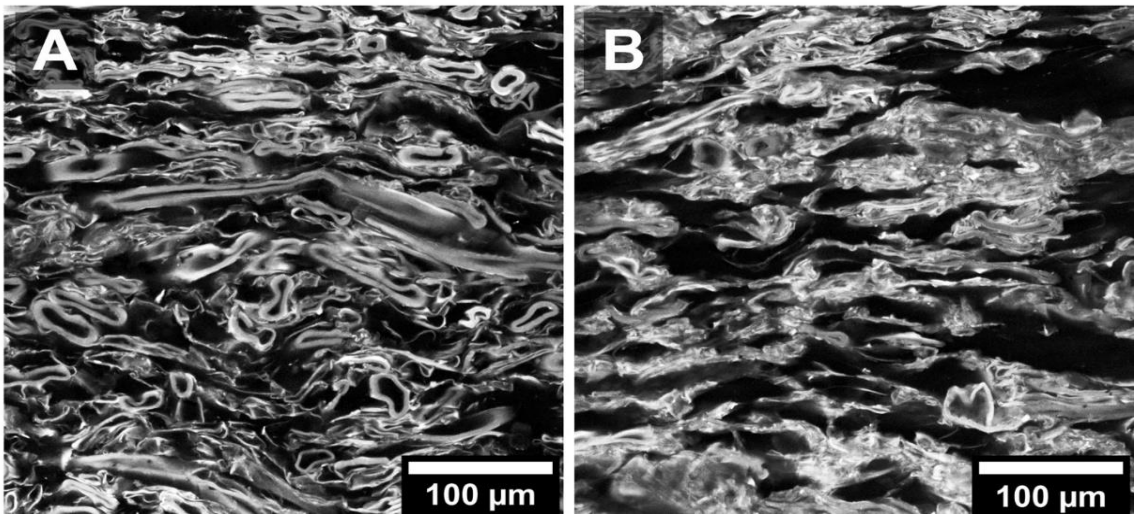

Fig. 3. Examples of CLSM images of compression molded PP composite panels reinforced with 50% cellulose fines by weight. Greyscale images with PP in black and cellulosic material grey/white. A) CTMP – relatively even distribution of cellulosic material; B) SMC fines – agglomerated distribution of cellulosic material

Based on density data (Fig. 2) and microscopical analysis (Figs. 3 and 4), there were no significant differences in density, layering, or gross agglomeration in the composites that would render them unsuitable for comparison.

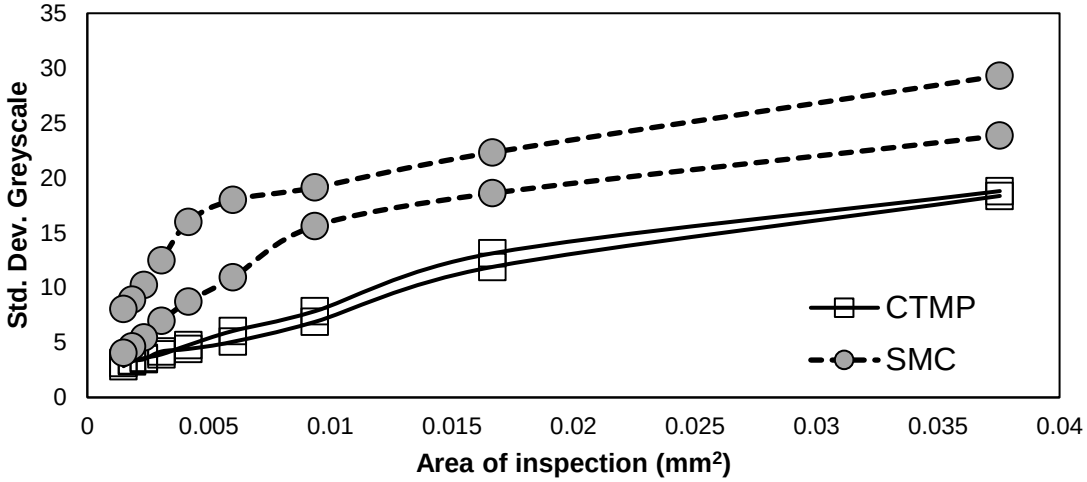

Fig. 4. Standard deviation in greyscale over a range of inspection areas**.** Only fiber and fines material are recorded as PP has no autofluorescence, and each cross section was divided up into a grid of squares with widths ranging from 40 to 200 µm.

Physical Properties of Wet-formed Compression Molded Composites

The modulus of elasticity (MoE) (Fig. 5) and modulus of rupture (MoR) (Fig. 6) both had a clear decreasing trend with the addition of fines material. The decrease was significantly greater for the CTMP fines than for the SMC fines, for both MoE and MoR.

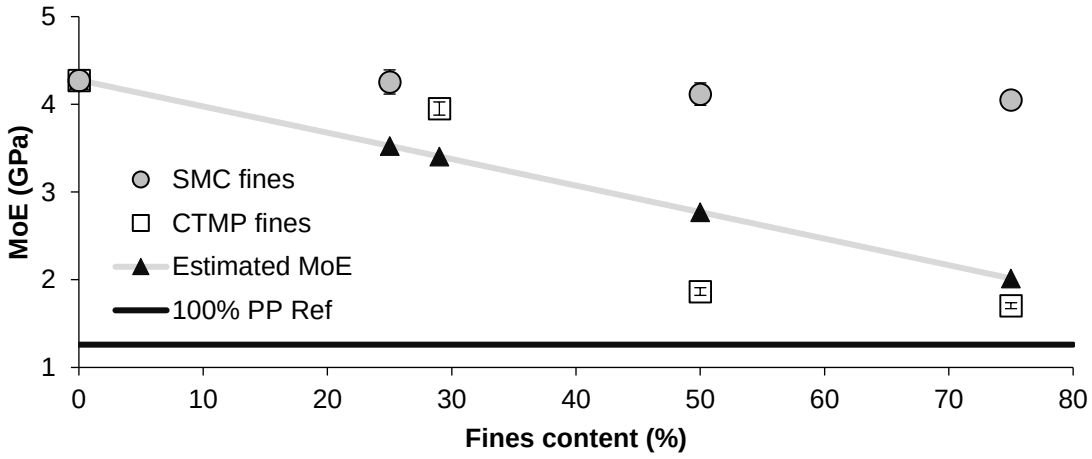

Fig. 5. Modulus of Elasticity (MoE), of compression molded composites with increasing fines content. A limited decrease was observed for fibrillar fines (75% SMC fines significantly lower MoE than 0% SMC fines), but a significant decrease was observed for blocky CTMP fines. The estimated MoE illustrates the linear regression between 100% fibers (at 40% loading) and 100% pure PP.

Figures 5 and 6 include the estimated linear regression, illustrating the expected reinforcement if the fines were not present in the composite.

For MoE, at 25 to 30% fines fraction of the cellulose content, there was a reinforcement above the estimated regression value for both CTMP and SMC fines. The CTMP fines stiffness was however already significantly lower than the observed SMC fines stiffness suggesting a statistically significant negative influence of CTMP fines at this

loading compared to SMC fines. The results for SMC fines more closely followed the trend expected for a cellulose filler material, and supports the observations by Fekete *et al*. (2018) with low aspect ratio cellulose fibers in thermoplastic starch and Toriz *et al*. (2002) with lignin particles in PP.

At higher fines loadings, the CTMP fines MoE reinforcement fell away almost completely. The reduction in properties was higher than expected. It does, however, support observations made by Gallagher and McDonald (2013) for the flexural stiffness of composites of very small wood particles in PE, and by Peltola *et al*. (2011, 2014) for pulp fines fractions of in PP and PLA.

Additionally, this observation supports the findings of Miettinen *et al*. (2012, 2015, 2016) that the fines fraction (near 50%) could be discarded from the modeling of the physical properties of polymer matrix wood fiber composites without a significant deviation of the modeled results from the experimental results, *i.e*., that the fines held no explanatory power of the composite reinforcement.

Potentially, this may indicate that the CTMP fines fraction is providing little or no additional contribution to stiffness beyond that already imparted by the long fiber fraction. The observed flexural stiffness of a sample containing in total 20% long fibers (50/50 fiber/fines) was 1.87 GPa, which corresponds well with Stark and Rowlands (2003) reported stiffness range of 1.8 to 1.9 GPa for 20% wood flour and fiber in PP.

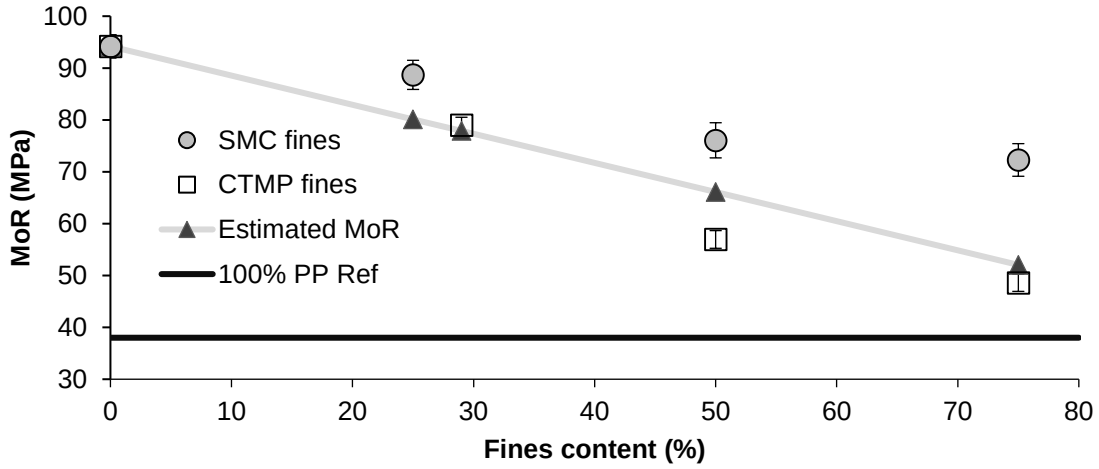

Fig. 6. Modulus of Rupture (MoR) of compression molded composites with increasing fines content. For both types of fines, a significant decrease in stiffness was observed with increasing fines content. The estimated MoR line and point illustrates the linear regression between 100% fibers (at 40% loading) and 100% pure PP.

For MoR, the observed trends were similar to the observed trends for MoE. In this case however, the trend for both fines materials more closely followed the estimated linear regression, which estimates the properties of the composite with the fines material fraction removed. This was particularly observed for the CTMP fines, indicating that they contributed little if any stiffness reinforcement to the composite.

This contradicts the observations made by Gallagher and McDonald (2013), who saw an increase in MoR with increasing fines content in PE, but supports the observations reported by Peltola *et al*. (2014) and Miettinen *et al*. (2015) for PP and PLA.

The results for compression molded PP composites indicate that fines have only

minor, if any, benefits as a reinforcing agent. Thus, fines removal from polymer matrix composites may be beneficial for composite physical reinforcement.

For CTMP fines, this result was interpreted as an effect of the reduction of the mean aspect ratio of the cellulose reinforcement. This was the result of an increasingly higher proportion of low aspect ratio fines particles. Furthermore, the effective aspect ratio for reinforcement of the CTMP may have been further decreased *via* agglomeration of the CTMP fines during the wet forming and compression molding process.

Conversely, for SMC fines the observation of a negative effect on MoR, with little or limited effect on the MoE, could indicate that more than just aspect ratio was affecting properties. It is well documented that fibril agglomeration has been a major limitation in the commercial utilization of micro-fibrillated cellulose in composites (Sandquist 2013). In the present study, there were likely insufficient mechanisms for the requisite dispersion of the SMC fines to achieve physical reinforcement of the composite.

CONCLUSIONS

1. In wet-formed compression-molded polymer matrix composites, both primarily blocky (CTMP) and fibrillar (SMC) fines had a significant negative impact on modulus of rupture (MoR). Additionally, blocky CTMP fines also had a significant negative impact on modulus of elasticity (MoE). It is postulated that this is due to fibril agglomeration, in the case of fibrillar fines, and low aspect ratio in combination with some agglomeration, in the case of blocky fines.
2. The results indicate that for compression molded polymer matrix composites, fines material that has not been well dispersed has limited reinforcing capability. Only negative trends where observed on most mechanical properties. This is consistent with modeling results shown for polymer matrix composites materials produced *via* injection molding.

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