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Size reduction of poplar wood using a lathe for biofuel manufacturing: effects of biomass crystallinity on sugar yield

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Keywords

Abstract

Poplar wood can be used as a feedstock for manufacturing cellulosic biofuel (ethanol) as an alternative to petroleum-based liquid transportation fuel. Producing biofuel from poplar wood involves reducing poplar wood into small particles (known as size reduction), hydrolyzing cellulose inside poplar particles to fermentable sugars, and converting these sugars to ethanol biofuel. Size reduction is usually done by wood chipping and biomass milling. In the literature, there are inconsistent reports about effects of particle size and biomass crystallinity on sugar

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yield (proportional to ethanol yield). An important reason for this inconsistence is that effects of these two biomass structural features (particle size and biomass crystallinity) on sugar yield are confounded with current size reduction methods. In this study, a lathe was used to produce poplar wood particles with (statistically) the same particle size (thickness) but different levels of biomass crystallinity, making it possible to investigate effects of biomass crystallinity on sugar yield without being confounded with effects of particle size. Results from this study show that, for the three levels of biomass crystallinity tested, sugar yield increased as biomass crystallinity decreased.

1 Introduction

Liquid transportation fuels currently used in the U.S. are mainly petroleum based (EIA, 2013a; EIA, 2013b; EIA, 2013c). In 2011, the U.S. transportation sector consumed about 18.95 million barrels of petroleum per day, and 45% of them were imported (EIA, 2013d). The dependence on foreign petroleum threatens the nation's energy security. Another issue of consuming petroleum-based transportation fuels is greenhouse gas (GHG) emissions. One-third of the total carbon dioxide emissions in the U.S. are from the use of petroleum-based transportation fuels (Greene et al. 2011).

Biofuels are critical to addressing these issues. Biofuels have the potential to reduce GHG emissions by as much as 86% (Wang et al., 2008). Because biofuels are made from plant-based feedstocks, the carbon dioxide released during combustion is "recycled" by plants as they grow (RFA, 2013). In addition, Cellulosic biofuels are produced from cellulosic biomass, including agricultural and forestry residues and dedicated energy crops. Unlike other types of feedstocks (e.g. corn, sugar cane, and soybean) for biofuels, cellulosic biomass does not compete with food production for the limited agricultural land (Zhang et al., 2011; Gray et al., 2006).

Major processes of biofuel manufacturing from poplar wood are listed in Figure 1. First, size reduction reduces the particle size of poplar wood (Lynd, 1996; Zhu et al., 2009a; Zhu et al., 2009b; Zhu et al., 2010). Pretreatment helps to make cellulose in the biomass more accessible to enzymes during hydrolysis. Hydrolysis depolymerizes cellulose into its component sugars (glucose). Afterwards, fermentation converts glucose into biofuel (ethanol) (Rubin, 2008; Houghton et al., 2006; von Sivers and Zacchi, 1996).

Size reduction of poplar wood is necessary because large-size woody biomass cannot be converted to biofuels efficiently with current conversion technologies (Wyman et al., 2009). Size reduction of poplar wood usually involves two steps. The first step is wood chipping. Machines available for wood chipping include disk, drum, and V-drum chippers (Wyman et al., 2009). The second step is biomass milling to further reduce the wood chips into small wood particles. This step is usually conducted on hammer mills (Zhu et al., 2009a; Negro et al., 2003), knife mills (Wyman et al., 2009; Nutor and Converse, 1991; Sierra et al., 2009), compression mills (Zhu et al., 2009a), or ball mills (Chang and Holtzapple, 2000).

Two important structural features of cellulosic biomass are biomass crystallinity and particle size (Zhu et al., 2008). Reported relationships between sugar yield and these two features are summarized in Table 1. It can be seen that reported relationships are inconsistent. Some researchers reported that lower biomass crystallinity produced higher sugar yield, while some other researchers did not support such a relationship. Inconsistent results are also reported for relationships between particle size and sugar yield as analyzed by Zhang et al. in their review paper (Zhang et al., 2013b).

An important reason for this inconsistence is that, with current size reduction methods, effects of these two features on sugar yield are confounded. Current size reduction methods tend

to change particle size and biomass crystallinity simultaneously. With current size reduction methods, in order to produce smaller particles, longer milling time is usually needed. In general, longer milling time also decreases biomass crystallinity by generating more impact and deformation to disrupt the crystalline structure of cellulose in the biomass (Zhu et al., 2008).

In this study, a metal-cutting machine (lathe) was used to produce poplar wood particles with (statistically) the same particle size (thickness) but different levels of biomass crystallinity. This effort made it possible to study effects of biomass crystallinity on sugar yield without being confounded with effects of particle size.

2 Experimental set-up and measurement procedures

2.1 Poplar wood material

The poplar wood used in this study was purchased from a local lumber company (Griffith Lumber Co., Manhattan, KS, USA). The size of the poplar lumber boards was 156 mm × 156 mm × 1,000 mm. As shown in Figure 2, poplar wood logs were cut from the lumber board using a hole saw (Milwaukee Electric Tool Co., Brookfield, WI, USA) with an inner diameter of 146 mm, on a drilling machine. Then the poplar wood log was fixed on a lathe (Monarch Machine Tool Co., Sidney, OH, USA) using a three-jaw chuck. A center hole with the diameter of 38.26 mm was drilled (using a twist drill mounted on the tailstock of the lathe) into the wood log to obtain the hollow cylinder workpiece. The inner surfaces of the hollow cylinders were machined by a boring tool to reduce the wall thickness (the distance between outer and inner radii of the hollow cylinder). The hollow cylinders were used for size reduction experiments.

2.2 Experimental set-up and conditions

The experimental setup is shown in Figure 3. Size reduction experiments were conducted on a lathe (Monarch Machine Tool Co. Sidney, OH, USA). The cutting tool was custom made with AISI T8 high speed steel. The tool geometry is shown in Figure 4. The rake angle of the tool could be adjusted by rotating the tool holder (NKLNR-121B, Kennametal Inc., Latrobe, PA, USA) along its axial direction. Eight slots (with 1 mm wide for each slot) were cut into the workpiece using a hacksaw, dividing the hollow cylinder workpiece into eight equal parts, as illustrated in Figure 5. The continuous chip would break (into particles) at locations of these slots. When the lathe spindle rotated one revolution, eight particles with the same length were produced. The experimental conditions are listed in Table 2. No coolant was used. A large white paper board was placed on the lathe bed to collect poplar wood particles. After the particles were collected, they were kept in zip bags and stored in a refrigerator at 4°C before further use.

2.3 Measurement of particle size

In this study, particle size is represented by particle thickness (a_0). It was measured using a caliper (IP-65, Mitutoyo Corp., Kawasaki, Japan), as shown in Figure 6. A typical particle was shown in Figure 7. Particles were curved when cut off from the wood cylinder workpiece, as shown in Figure 7(a). If they were manually flattened, they would look like the one shown in Figure 7(b). Thirty particles under each condition were randomly picked for measurement of particle thickness.

2.4 Measurement of biomass crystallinity

Cellulose in cellulose biomass consists of amorphous regions and crystalline regions, as illustrated in Figure 8. Biomass crystallinity is used to describe the percentage of crystalline regions of cellulose and expressed as crystallinity index (CI) (Sunkyu et al., 2010). A higher CI

means that cellulose in cellulose biomass has a higher percentage of crystalline regions. It has been suggested that amorphous regions of cellulose degrades more easily than crystalline regions (Puri, 1984; Fan and Beardmore, 1980). Therefore, a higher CI would result in lower enzyme accessibility, and, hence, lower sugar yield. CI was measured by an X-ray diffractometer (MiniFlex II, Rigaku Americas Corp., The Woodlands, TX, USA) and calculated using analysis software PDXL (Version 1.6.0.0, Rigaku Americas Corp., The Woodlands, TX, USA). For each test condition, three particles were randomly picked for CI measurement. For each measurement, one poplar particle was placed on the sample holder of the X-ray diffractometer.

2.5 Measurement of sugar (glucose) yield

Prior to sugar yield measurement, collected poplar particles were treated using dilute acid pretreatment. The pretreatment was carried out in the 600-ml reaction vessel of a Parr pressure reactor (4760A, Parr Instrument Co., Moline, IL, USA). Poplar particles were mixed with diluted sulfuric acid to obtain biomass slurry with 5% solid content (10 g of poplar particles in 200 mL of 2% diluted sulfuric acid). Pretreatment time was 30 min, and pretreatment temperature was 140 °C.

After pretreatment, biomass was washed three times with 300 mL of hot deionized water (85 ºC) using a centrifuge (PR-7000M, International Equipment Co., Needham, MA, USA). The rotation speed of the centrifuge was 4,500 rpm. The purpose of biomass washing was to remove acid residues and inhibitors (substances that would bind to enzymes and decrease their activity to depolymerize cellulose to glucose) formed during pretreatment.

Then, the pretreated biomass was processed by enzymatic hydrolysis, following the National Renewable Energy Laboratory (NREL) analytical procedure (Selig et al., 2008). Enzyme complex Accellerase 1500[TM] (Danisco US Inc., Rochester, NY, USA) was used in the sodium

acetate buffer solution (50 mM, pH 4.8) with 0.02% (w/v) sodium azide to prevent microbial growth during hydrolysis. Enzymatic hydrolysis was carried out in 125-mL flasks with 50 mL of slurry in a water bath shaker (C76, New Brunswick Scientific, Edison, NJ, USA) with the agitation speed of 110 rpm at 50 °C for 72 hours. The dry mass content of the hydrolysis slurries was 5% (w/v) and the enzyme loading was 1 mL/g of dry biomass. After 72 hours of enzymatic hydrolysis, the hydrolysis slurries were sampled by withdrawing 0.1 mL of slurry from each flask. Sample slurries were then mixed with 0.9 mL of double-distilled water in 1.5-mL vials. The vials were placed into boiling water for 15 min to deactivate the enzyme. Then, the sample slurries were centrifuged in a micro centrifuge (RS-102, REVSCI Co., Lindstrom, MN, USA) at 10,000 rpm for 15 min. The supernatants were filtered into 2-mL autosampler vials through 0.2-μm syringe filters (EMD Millipore Corp., Billerica, MA, USA). The filtered samples in the autosampler vials were ready for sugar analysis.

Sugar analysis was done using a high performance liquid chromatography (HPLC) system (Shimadzu, Kyoto, Japan) equipped with an RPM-monosaccharide column (300 × 7.8 mm; Phenomenex, Torrence, CA, USA) and a refractive index detector (RID-10A, Shimadzu, Kyoto, Japan). The mobile phase was 0.6 mL/min of double-distilled water, and oven temperature was 80 °C. HPLC can identify and quantify individual components of a liquid mixture.

Sugar yield represents the amount of glucose produced from cellulosic biomass in enzymatic hydrolysis. A higher sugar yield means that more glucose is obtained. In this paper, sugar yield was determined by the following equation:

$$\text{Sugar yield} = \frac{G_{EH} \times V}{M_{EH}} \times 100\% \tag{1}$$

where G_{EH} is the glucose concentration (g/L) of slurry in the flask after hydrolysis, M_{EH} is the dry weight (g) of cellulosic biomass loaded in the flask before enzymatic hydrolysis, V is the total volume (L) of slurry in the flask in enzymatic hydrolysis.

3 Results and discussion

Results on particle size (thickness) are shown in Figure 9. For particles produced with different tool rake angles, there are no significant differences in particle sizes (thickness). Error bars for each data point in Figure 9 (and Figures 11-13) were drawn using the 95% confidence intervals of the means. Means of data for each response variable under different experimental conditions were compared by one-way analysis of variance (ANOVA) using software Minitab (Version 15, Minitab, Inc., State College, PA, USA). The following assumptions are used: (a) response variables are normally distributed; (b) samples are independent; and (c) variances of populations are equal.

Figure 10 illustrates particle formation in orthogonal cutting. The cutting edge of a wedge-shaped tool is perpendicular to the cutting direction. As the tool is forced into the workpiece material, the particle (chip) is formed by shear deformation along a shear plane oriented at an angle φ (shear angle) with the workpiece surface. Along the shear plane, plastic deformation of the workpiece material occurs. Shear angle is an indirect measure of the deformation severity of the produced particles. Shear angle φ is determined by Equation (2) (Boothroyd and Knight, 2006):

$$\tan \varphi = \frac{\dfrac{a_c}{a_0} \cos \gamma_0}{1 - \dfrac{a_c}{a_0} \sin \gamma_0} \tag{2}$$

where a_c is the uncut particle thickness, a_0 is the particle thickness, γ_0 is the rake angle of the tool. The uncut particle thickness a_c was the thickness of the layer of the workpiece material being removed per revolution of the workpiece. In this experimental setup, a_c was determined by the feedrate (mm/r). According to metal cutting theory (Singal and Singal, 2009; Juneja and Seth, 2003), when cutting with a tool that has a larger rake angle, the workpiece material undergoes less severe deformation. When being cut with a tool that has a smaller rake angle, the material undergoes more severe deformation. As shown in Figure 11, shear angle increased when tool rake angle increased from 20° to 30°. A smaller tool rake angle would produce a smaller shear angle, and cause more severe deformation in produced particles.

Figure 12 shows that biomass crystallinity index (CI) decreased as tool rake angle decreased. This observation could be explained as follows: when a smaller tool rake angle was used, larger cutting force would be applied onto the workpiece material and particles would undergo more severe deformation. This could cause crystalline regions in cellulose to deform and transform into amorphous regions (Fan and Beardmore, 1980). Therefore, CI decreased.

It is important to note that there was no significant difference in particle thickness when CI changed, as shown in Figure 12. Since the surface area of particles was well controlled as a constant by the slots cut into the hollow wood cylinder workpiece, biomass particles with different crystallinity but the same particle size were produced. Lower biomass crystallinity was not associated with smaller particle size. Therefore, the confounding effects of particle size and biomass crystallinity were separated.

Results on sugar yield are shown in Figure 13. It can be seen, from Figures 12 and 13, that sugar yield increased as biomass crystallinity decreased.

The authors have also studied effects of particle size on sugar yield independently without being confounded with biomass crystallinity (Zhang et al., 2013a). In that study, poplar wood particles with different levels of particle size but the same biomass crystallinity were produced using a lathe. Experimental results show that sugar yield increased as particle size became smaller.

4 Conclusions

This study demonstrated an approach to separate confounding effects of particle size and biomass crystallinity. Hence, it became possible to investigate effects of biomass crystallinity on sugar yield independently. The following conclusions can be drawn.

1) Poplar wood particles produced with different tool rake angles had (statistically) the same size (thickness).

2) Poplar wood particles produced with different tool rake angles had different biomass crystallinity. Biomass crystallinity decreased as tool rake angle decreased.

3) For the three levels of biomass crystallinity tested in this study, sugar yield increased as biomass crystallinity index became smaller.

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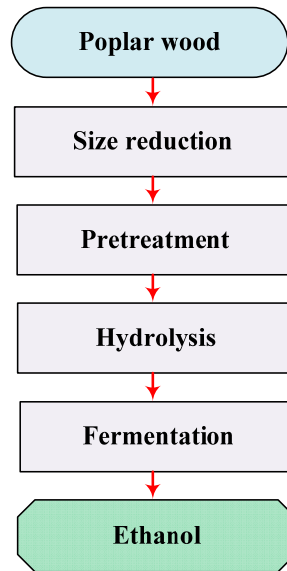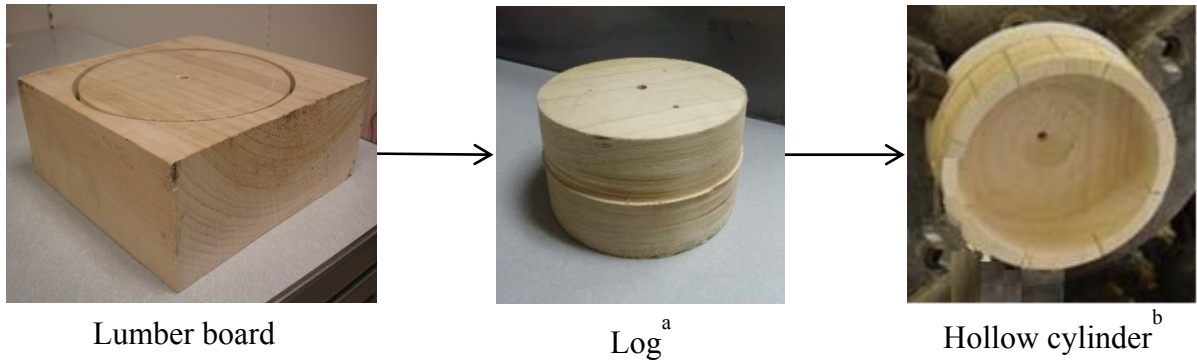
Figure 1 Major processes of biofuel manufacturing from poplar wood (after [14])

| Lumber board | Log[a] | Hollow cylinder[b] |

a.
   A poplar wood log was cut by a hole saw from a lumber board. Since the maximum cutting depth of the hole saw was smaller than the thickness of the lumber board, the wood log was produced after two cuts, each cut from each side of the lumber board.

b.
   A center hole was first drilled using a twist drill mounted on the tailstock of the lathe. The inner surface of the hollow cylinder was machined by a boring tool.

Figure 2 Poplar wood workpiece preparation
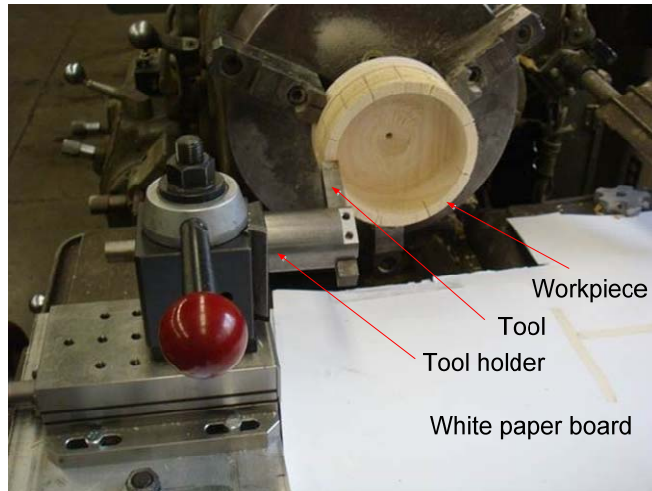
Figure 3 Experimental setup
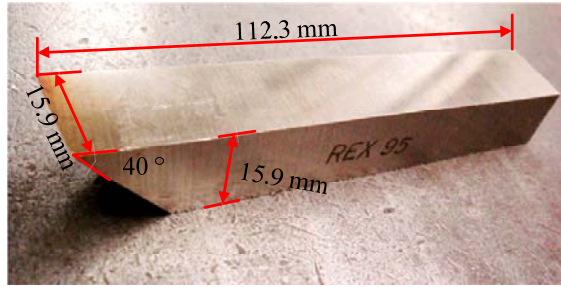
Figure 4 Dimensions of the cutting tool.

Right view   Front view
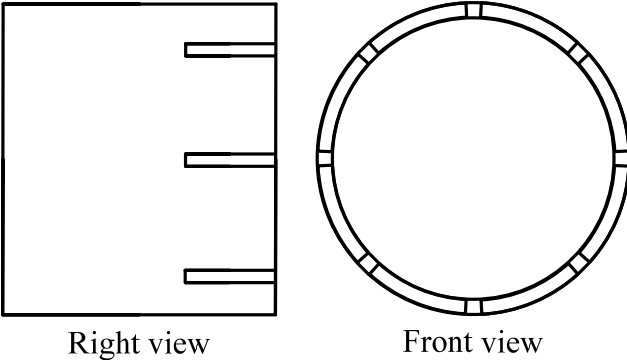
Figure 5 Illustration of eight slots on the workpiece
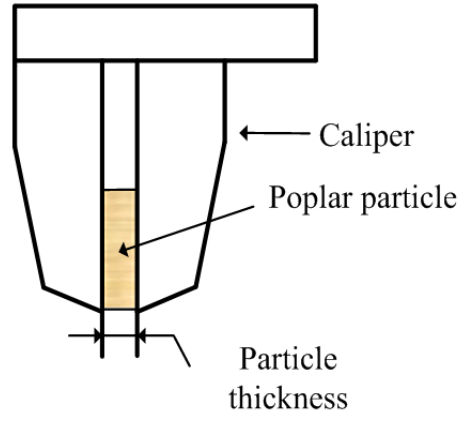
Figure 6 Illustration of poplar particle thickness measurement (not to scale)

(a) After cutting   (b) After flattening
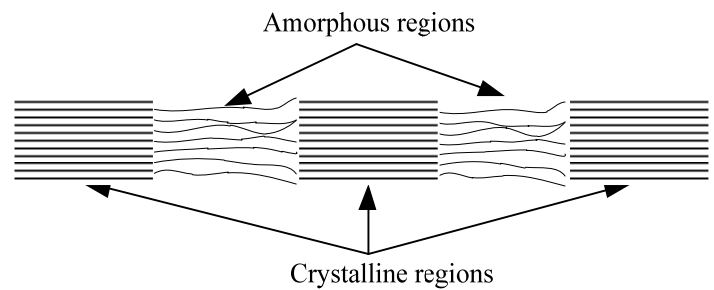
Figure 7 Pictures of a poplar particle

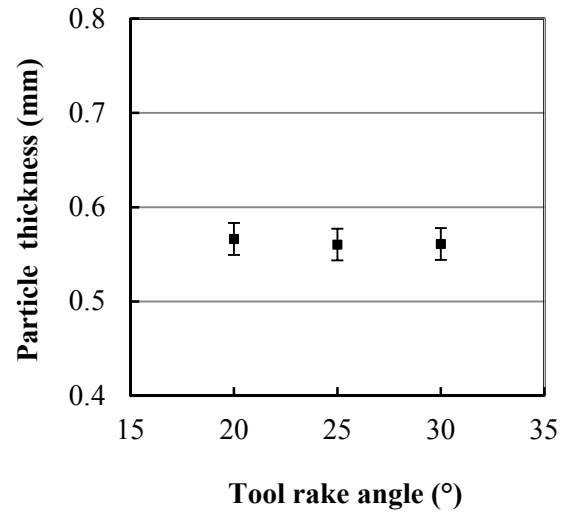Figure 8 Amorphous and crystalline regions in cellulose (after [3])

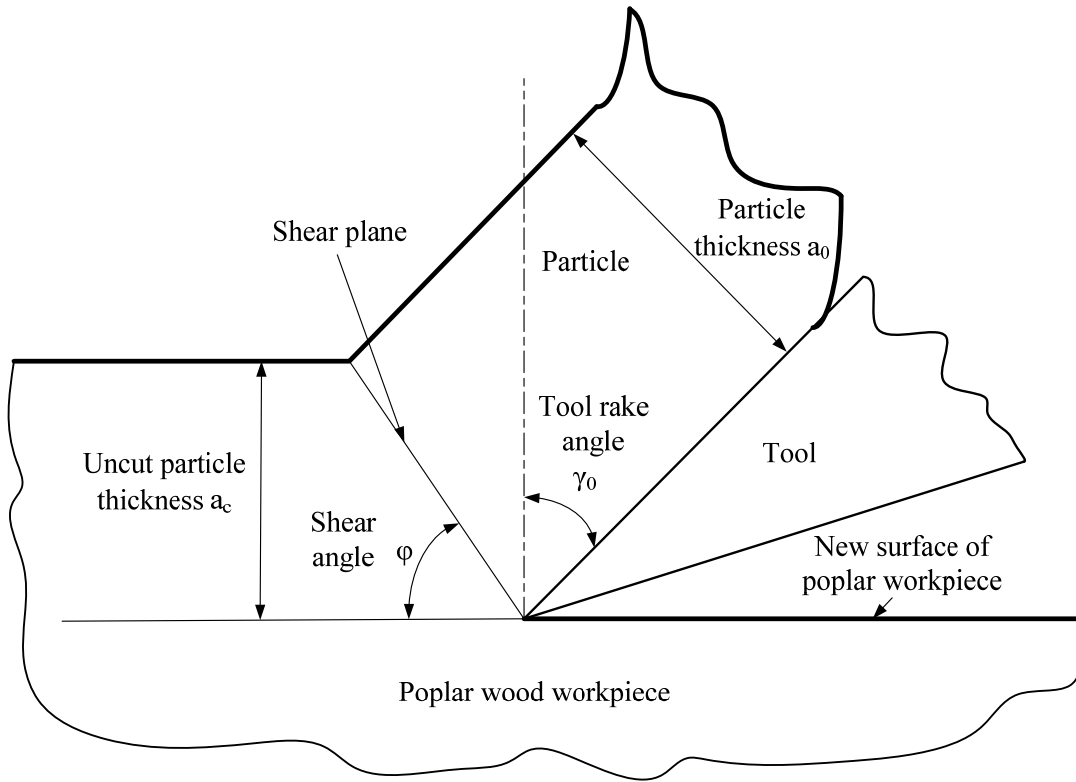Figure 9 Effects of tool rake angle on particle thickness

Shear plane

Particle

Particle thickness a_0

Uncut particle thickness a_c

Tool rake angle γ_0

Tool

Shear angle φ

New surface of poplar workpiece

Poplar wood workpiece

Figure 10 Illustration of particle formation in orthogonal cutting (after (Boothroyd and Knight, 2006))
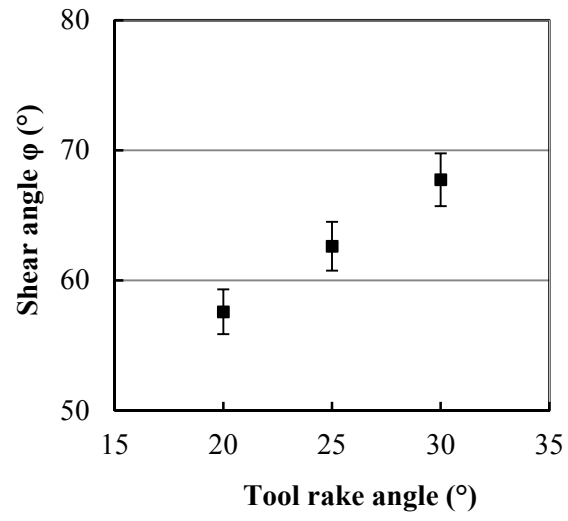
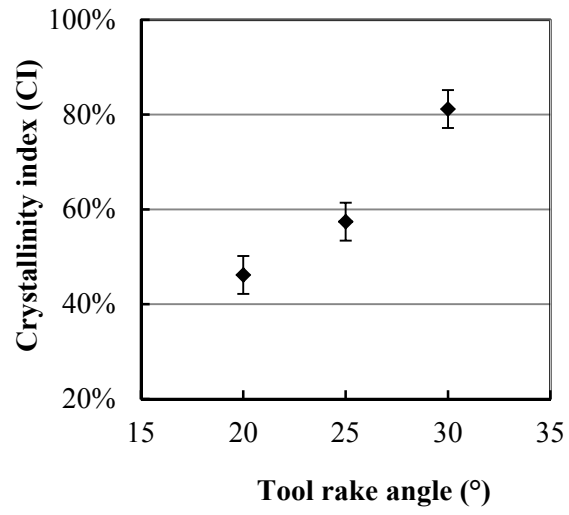Figure 11 Effects of tool rake angle on shear angle

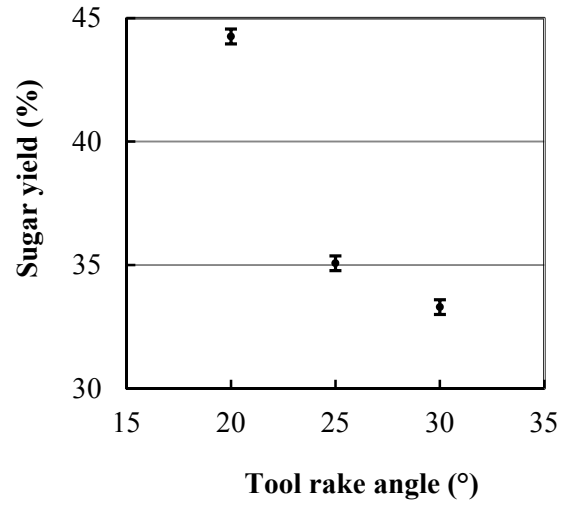Figure 12 Effects of tool rake angle on crystallinity index

Figure 13 Effects of tool rake angle on sugar yield

Table 1 Reported relationships between structural features and sugar yield

Structural feature	Relationship between structural feature and sugar yield	Reference
Biomass crystallinity	As biomass crystallinity decreased, sugar yield increased.	(Chang and Holtzapple, 2000; Sinitsyn et al., 1991; Fan et al., 1981; Koullas et al., 1990; Caulfield and Moore, 1974)
	No correlation.	(Puri, 1984; Grethlein, 1985; Gharpuray et al., 1983)
Particle size	As particle size decreased, sugar yield increased.	(Chang and Holtzapple, 2000; Grethlein, 1985; Gharpuray et al., 1983; Klass, 1998)
	No correlation.	(Sinitsyn et al., 1991; Draude et al., 2001)

Table 2 Experimental conditions

Process variable	Value
Tool rake angle	20°, 25°, 30°

Cutting speed = 4.0 m/s;
Feedrate = 0.508 mm/r;
Depth of cut = 8.67 mm.