



Recirculating calcium hydroxide solution: A practical choice for on-farm high solids lignocellulose pretreatment



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ABSTRACT

Pretreatment is considered a necessary step in the use of lignocellulosic biomass for biochemical conversion to higher value products. There are multiple choices of chemicals for pretreatment in industrial settings, however on-farm choices are constrained to those that function well at near ambient conditions with minimal specialized equipment, personnel training, and require limited waste disposal. This work presents a novel pretreatment system biomass using a recirculating, saturated calcium hydroxide (lime) solution in an up-flow, high solids (14–16% w/w) configuration at ambient conditions. In this system, lime solids were efficiently consumed, post-pretreatment washing of substrate was not required, and energy and resources were conserved. Pretreatment effectiveness was assessed by glucose yield comparisons for both switchgrass and corn stover. Based on mean glucose yields from 5 mm corn stover, lime pretreatment would require 350 kg of dry stover to produce 100 kg glucose at a chemical cost of \$8.67 while NaOH pretreatment would require 300 kg of chemical at a cost of \$22.38.

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1. Introduction

Biomass transport is one of the most costly processes in bio-fuel production; biochemical conversion on farm would allow for the harvesting, storage, and conversion to be completed in the same location (Klein-Marcuschamer and Blanch, 2015). However a successful on-farm conversion process requires optimization of all stages of operation: storage, size reduction, and pretreatment must be integrated.

The vast majority of studies related to the chemical pretreatment of lignocellulose have been conducted at the laboratory scale with conditions such as elevated temperatures and pressures, finely ground particles sizes, and with chemicals and processes that produce wastes which require careful disposal (Kumar et al., 2009). While these types of processes can produce an effective pretreatment, they are impractical or too expensive for a simple on-farm process. A practical on-farm process would be conducted under ambient conditions, using a relatively safe, inexpensive chemical which can be recovered, minimizing waste by-products, while achieving a reasonable level of pretreatment.

The use of calcium hydroxide (lime) as a lignocellulosic pretreatment chemical is not a new idea; it has been evaluated in many

studies (Ayeni et al., 2013), resulting in recommended pretreatment temperatures of 100–120 °C with treatment periods defined in hours (Chang et al., 1997), or for temperatures of 50–60 °C with the treatment periods defined in days or weeks (Xu et al., 2010). More recent work has re-examined the performance of lime at ambient temperatures (Rodrigues et al., 2016) and even below ambient temperatures (Khor et al., 2015), however in general these studies concluded that high temperatures were necessary to achieve sufficient glucose yield (Carey, 2014). In addition, these studies autoclaved the biomass prior to pretreatment; a practice which is impractical on-farm.

Previous studies did not adjust the process for $\text{Ca}(\text{OH})_2$'s limited solubility in water. $\text{Ca}(\text{OH})_2$ exhibits an interesting property that the solubility actually decreases with increasing temperature. For example, at 20 °C, 1.65 g/L of $\text{Ca}(\text{OH})_2$ will dissolve into solution, whereas only 0.071 g/L will dissolve into solution at 100 °C (Association, 2007); contrast this with the high temperature recommendation for $\text{Ca}(\text{OH})_2$ pretreatment and a logical disconnect is apparent. Lime's low solubility produces a far less aggressive alkali solution by limiting the hydroxyl ion concentration available in solution, which likely accounts for the longer recommended pretreatment period (Yan et al., 2015). The reviewed literature commonly recommended a static lime loading rate of 0.10 g/g dry matter (10% w/w) (Yan et al., 2015).

Undissolved $\text{Ca}(\text{OH})_2$ solids are problematic because they will deposit on the substrate instead of performing the desired

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Fig. 1. a) Vessel used for up-flow treatment showing in-flow port on the bottom of the vessel and exit port on the top. b) Stand showing six vessels in use simultaneously during high solids recirculating $\text{Ca}(\text{OH})_2$ pretreatment.

pretreatment. The unreacted $\text{Ca}(\text{OH})_2$ on the biomass also necessitates significant neutralization prior to hydrolysis, typically requiring large volumes of wash water or chemical neutralization with acids (Rodrigues et al., 2016). Pretreatment processes using alkali solutions result in final pH values typically above 10; whereas pH levels acceptable for enzymatic hydrolysis are typically 4.8–5.5. Neutralization methods used in $\text{Ca}(\text{OH})_2$ pretreatment studies include washing the solids with de-ionized water (Yan et al., 2015), adding organic or mineral acids to the solids (Falls and Holtzapple, 2011), or using gaseous carbon dioxide to reduce pH and enable the recovery of calcium as calcium carbonate (Park et al., 2010).

The biomass solids content of the pretreatment step impacts all aspects of the conversion process including water use, materials handling, and ultimately process economics (Modenbach and Nokes, 2012). The majority of pretreatment studies have been conducted with initial dry matter solids loadings ranging between 5% and 10% (w/w). A process is considered high solids at or greater than 15% (w/w); this demarcation signifies the transition from a slurry to stackable solids (Modenbach and Nokes, 2012). The use of high solids may offer economic advantages through improved efficiencies, however the lack of free water may slow chemical reactions, increased viscosity complicates material handling and mixing, and there is a potential to produce inhibitory compounds at higher concentrations (Rodrigues et al., 2016).

1.1. Objectives

The overall goal of this research was to determine whether $\text{Ca}(\text{OH})_2$ is an effective pretreatment when the solubility limitations of $\text{Ca}(\text{OH})_2$ are accounted for in the pretreatment design. The second objective of this work was to test the hypothesis that no

neutralization of the substrate would be needed if $\text{Ca}(\text{OH})_2$ solids did not deposit on the biomass, because only solubilized $\text{Ca}(\text{OH})_2$ was circulated through the biomass.

2. Materials and methods

2.1. Feedstock

The substrates used for this work were *Zea mays* (corn stover) and *Panicum virgatum* (switchgrass). The corn stover was Becks 6175 hybrid, harvested in the fall of 2013 at the C. Oran Little Research Center in Woodford County, KY. The Alamo switchgrass

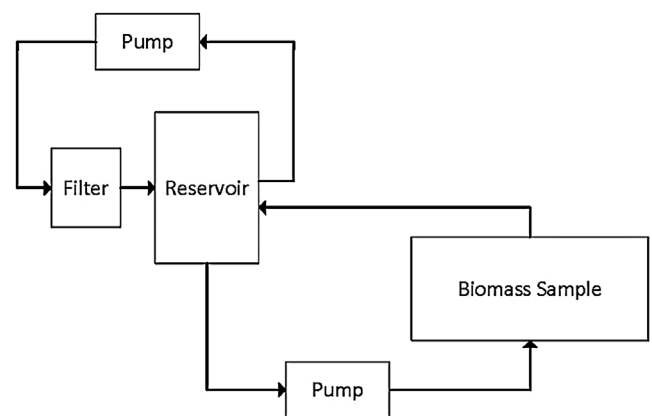


Fig. 2. Experimental pretreatment system schematic showing recirculating system for the saturated lime solution. The filter retained $\text{Ca}(\text{OH})_2$ solids, followed by the reservoir where the saturated $\text{Ca}(\text{OH})_2$ solution was stored.

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