Online Residence Time Distribution Measurement of Thermochemical Biomass Pretreatment Reactors at Various Conditions

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ABSTRACT

Residence time is a critical parameter that strongly affects the product profile and overall yield achieved from thermochemical pretreatment of lignocellulosic biomass during production of liquid transportation fuels. The residence time distribution (RTD) is one important measure of reactor performance and provides a metric to use when evaluating changes in reactor design and operating parameters. An inexpensive and rapid RTD measurement technique was developed to measure the residence time characteristics in biomass pretreatment reactors and similar equipment processing wet-granular slurries. Sodium chloride was pulsed into the feed entering a 600 kg/d pilot-scale reactor operated at various conditions, and aqueous salt concentration was measured in the discharge using specially fabricated electrical conductivity instrumentation. This online conductivity method was superior in both measurement accuracy and resource requirements compared to offline analysis. Experimentally measured mean residence time values were longer than estimated by simple calculation and screw speed and throughput rate were investigated as contributing factors. A semi-empirical model was developed to predict the mean residence time as a function of operating parameters and enabled improved agreement.

INTRODUCTION

Thermochemical pretreatment is a key step in the overall process of refining lignocellulosic biomass into sugars and ultimately alcohol or hydrocarbons for transportation fuels. Dilute acid-catalyzed thermochemical pretreatment physically disrupts the biomass structure and hydrolyzes most of the hemicellulose fraction and some of the cellulose fraction into fermentable sugar monomers prior to enzymatic hydrolysis of the remaining cellulose^{1,2}. One key chemical reaction is hydrolysis of xylan to the desired intermediate, xylose monomer, which further degrades to fermentation-inhibiting furfural and other byproducts as the reaction proceeds. Xylan hydrolysis reaction rate is primarily controlled by acid concentration and temperature, and the extent of reaction is governed by residence time. Since the desired monomeric sugar is an intermediate reaction product, effective reactor residence time control is necessary to target reactor conditions that favor high sugar yield.

Bench scale pretreatment experimentation is typically performed using batch reactors in which system residence time is carefully controlled within a narrow window by application of steam and quick depressurization to quench the reactions³. As pretreatment is scaled to the pilot plant and beyond, continuous flow reactors are preferred for plant operations and cost⁴. Residence time in continuous reactors is controlled by various mechanical conveyance means, including variable speed auger-screws within horizontal tubes. Continuous reactors generally have imperfect flow characteristics, where undesired back mixing and flow stratification lead to variations in residence time from those predicted from theoretical plug flow⁵. This variation is characterized by the residence time distribution (RTD) that describes the probability that a particle or chemical species will spend a specific time at desired reaction conditions. Maximal

xylose yield is achieved at a specific residence time, but non-ideal flow inside continuous pretreatment reactors distributes species about the targeted reaction time, which ultimately reduces the desired product yield⁶. A continuous reactor with a sharp, low-variance RTD with the mean residence time value at the target reaction time is favorable for translating from bench-scale batch reactor conditions and allows more precise control of reaction extent and ultimately the ability to maximize desired intermediate product yield. It is desirable to measure the RTD of these pretreatment reactors to verify residence time and also to enable study of main factors affecting RTD control.

Recent studies have reported measurements of the residence time in a biomass-fed pyrolysis reactor⁷ and screw conveyor⁸. Although these papers have brought valuable information to the literature for biomass processing equipment, the methods did not permit measurement at live reaction conditions. *Xi et. al.* measured RTD in a biomass pyrolysis reactor using the impulse response method by injecting a tracer and measuring the amount of tracer in samples collected over regular intervals. It is important to note, however, that this method was employed in an ambient environment instead of at real reaction conditions. This is suitable only if system rheology remains the same at ambient conditions and during the course of actual reaction. In the case of biomass pretreatment to sugars, the slurry rheological characteristics change dramatically during pretreatment and measurement of RTD at reaction conditions is absolutely necessary for accurate results. *Nachenius et. al.* measured the RTD of pine chips, rice, and sand in a horizontal screw conveyor at ambient conditions via step-change experiments by stopping the inlet flow and observing the flow response of the outlet by collecting samples at regular intervals. This equipment is similar to pretreatment reactors; however, when at operating conditions, the inlet

biomass flow may not simply be stopped without process upset. Steam is injected to rapidly heat the incoming feedstock, drive the reaction, and also compensate for heat loss. A step-change approach stopping all feed streams would confound RTD measurement as a constant slurry composition could not be maintained. Furthermore, it is unclear if deviating from steady-state flow in the reactor will provide a valid RTD for the pretreatment reactor, as fill fraction may affect the RTD itself. Finally, explosive flashing of biomass slurry at the reactor outlet presents difficulty and a serious safety hazard for manual sample collection to make use of offline analytical methods previously presented. The ability to perform *online* RTD measurements in a biomass thermochemical pretreatment reactor at live, steady-state reaction conditions is critical.

The online RTD measurement methods reported in the literature focus on polymer screw extruders⁹⁻¹¹ and food processing equipment^{12,13}. These methods include ultrasonic, photo-detected dye, magnetic, and radiofrequency identification (RFID) techniques. The machinery used in these examples is similar to pretreatment reactors, but the inhomogeneity, elevated temperature and pressure, and chemistry of pretreatment conditions present special challenges for the implementation of these online measurement techniques. For example, the RTD of chicken chowmein inside an ohmic heater sterilization system was measured online using RFID by *Tulsiyan et. al.* as a more accurate alternative to the industry-accepted magnetic method. Unfortunately, the RFID and magnetic methods do not translate well to biomass pretreatment reactors because RFID tags cannot withstand temperatures above 150°C and both RFID tags and magnets would likely suffer mechanical damage during screw conveying. Acid pretreatment reactors are primarily constructed of high-nickel alloys for corrosion resistance and do not readily attract magnets as they are paramagnetic. However, several minor parts are cold-worked

316L stainless steel, which exhibits some ferromagnetism and would capture magnets fed through the system and render the magnetic RTD detection method ineffective. *Lee et. al.* presented an online RTD measurement method for a polymer extruder involving a calcium carbonate tracer detected using ultrasound. This required the homogeneous polymer melt to completely fill the space between the emitting and receiving transducers. Unfortunately, pretreated biomass is a heterogeneous wet-granular slurry consisting of moist variable-sized biomass particles with steam-filled voids that renders the ultrasonic RTD method also ineffective. Photo-detection is another attractive method, but again suffers from the incompatibility of cameras/photo-sensors with reactor conditions involving high temperature and condensing steam.

This study provides a novel, rapid online RTD measurement method suitable for the harsh and non-ideal conditions within biomass pretreatment reactors where RTD measurement techniques previously reported would fail to perform. Electrical conductivity-based quantification of sodium chloride tracer was developed into a fast online detection technique that is applicable to pilot- and full-scale biomass pretreatment reactors and also other processes that involve high solids aqueous slurries such as wood pulping and food processing, to name a few. A pilot-scale thermochemical pretreatment reactor served as a platform for demonstrating this method and is analogous to other pilot- and full-scale reactors. The method developed in this work is applicable beyond biomass pretreatment to any process where electrical conductivity can be manipulated and measured online at process conditions.

MEASUREMENT METHODOLOGY

Our RTD measurement method was developed working with a Metso, Inc. (Norcross, GA, USA) continuous pilot-scale horizontal auger-tube pretreatment reactor used for research activities¹⁴. This Hastelloy C-2000 constructed reactor, represented in Figure 1, was maintained at 160°C by saturated steam injection. Tub ground and hammer milled (with a 19 mm rejection screen) corn stover was pre-wetted with 1.0 wt% sulfuric acid to attain ~50 wt% total solids content and was fed into the reactor at a rate of 600 dry kg/d. A small amount of liquid was involuntarily pressed from the wetted feedstock within the tapered screw feeder and was discarded. However, additional fresh water was injected inside the shredder conveyor to maintain a reactor exit total solids content of 28%.

Figure 1. 600 kg/d pretreatment reactor to-scale elevation view of components with respective operating parameters during method validation. Reactor tube 1 and 2 speeds are variable.

Theoretical plug-flow residence time within the entire reactor (t_{th}) was calculated by summing individual component residence times using

$$t_{th} = \sum \frac{V_i}{v_i}, \quad (1)$$

where V is the active volume inside each tube (cm^3) and v is the volumetric flow rate of each auger screw (cm^3/min); also, v is equivalent to screw speed (rev/min) $\times$ pitch (cm) $\times$ cross-

sectional area (cm^2). These sections were operated starved with no more than 44% volumetric fill. The impregnator section (component C in Fig. 1) was assumed a poor conveyor due to its vertical orientation, and component residence time was calculated independent of auger speed by substituting Q , volumetric biomass slurry flow (equal to mass flow / mean bulk density), in place of v . Feedstock bulk density was 80 and exiting slurry was typically 130 dry kg/m^3 , measured using standard industrial practices¹⁵, and the mean was taken about these two. Reactor tube 2 discharge chute residence time was assumed negligible and theoretical residence time was calculated at 10.7 min for conditions listed in Fig. 1.

Sodium chloride was selected as a tracer compound for biomass as it is inert at reaction conditions and is readily quantified using electrical conductivity. This process operates at sufficient solids concentration to produce non-weeping wet-granular slurries (i.e. solid-phase bulk flow without separate liquid) and it was assumed the sodium chloride dissolved directly into the liquid associated with the porous biomass particles. Impulses of fine granular sodium chloride tracer were introduced over the course of ≤ 1 s into reactor feed at the tapered screw feeder (approaching an ideal Dirac-delta function for impulse response type RTD measurement) while the reactor was at normal steady state conditions, and exit tracer concentration was measured versus elapsed time. Dosing in the range of 50–200 g NaCl (2–8 g per kg dry solids/h) was assessed and signal saturation was not observed across this range when operating at 28% total solids fraction in the reactor. An optimum dose was reached at 6 g NaCl/kg dry solids/h, which maximized the signal response over baseline while moderating additional torque load placed on the tapered screw feeder from ingestion of incompressible solids.

Figure 2. Cutaway diagram and photograph of finished conductivity probes. Pipe thread size is 3/4" NPT (2.5 cm OD). O-rings (black circles) are used to seal and electrically insulate the Hastelloy electrode inside the ceramic insulator and Hastelloy pipe plug.

NaCl concentration in the reactor exit slurry was quantified by measurement of apparent electrical conductivity (without compensation for temperature or discontinuous electrical paths formed by gaseous voids within the wet-granular slurry) in the discharge airlock immediately before flashing and quenching the pretreatment reaction. Electrical conductivity-based quantification was particularly advantageous because a three-dimensional slurry sample was measured by this method and offered more uniform and robust analysis in case of imperfect tracer mixing with process slurry. Cycling airlock dischargers are typical of continuous pilot scale pretreatment reactors and, in this case, consisted of two ball valves between a short vertical DN100 Hastelloy C-2000 pipe as shown in Figure 1. This airlock received slurry with the top valve open and bottom valve closed until the end of each cycle, when the top valve momentarily closed and the bottom valve opened to dump and flash slurry to atmospheric conditions. Special

probes, illustrated in Figure 2, were constructed of a Hastelloy C-276 electrode and 3/4" NPT pipe-plug body, FKM o-ring seals, and insulator made of Macor (Corning, Inc, New York, NY, USA) machinable ceramic. Two of these probes were installed facing one another through opposite sides of the vertical airlock pipe as shown in Figure 3 with a probe tip gap of 8.3 cm and electrode face diameter of 1.4 cm. Probe tips extended 0.8 cm from the pipe inner wall and steam cleaning jets installed above the probes were automatically pulsed every discharge event to prevent slurry from clinging and stagnating on the tips. Different reactor designs and process slurry characteristics could eliminate the need for steam jets. We should also note that larger scale reactors are known to use continuous variable-position discharge valves instead of cyclical airlocks. In this case, the electrode probes could be positioned just upstream of the discharge valve and would be exposed to fresh slurry just before exiting the reactor.

Figure 3. Installation detail of conductivity probes (highlighted) in lower threaded ports of discharge valve airlock pipe. Cleaning steam pulsed through nozzles pointing downward from upper pair of threaded ports. Automated ball valves are located at the top and bottom of the airlock.

An Omega Engineering, Inc. (Stamford, CT, USA) CTDX-90-1P analyzer measured the apparent conductivity across the reactor probes. Measurements were not compensated for temperature: NaCl conductivity varies linearly with temperature between 0–200°C and is not affected by pressure below 400 MPa¹⁶. The electrode cell constant in the reactor was determined using a NaCl conductivity reference solution in a dimensionally identical 316L stainless steel test cell. Bench tests with NaCl-doped pretreated corn stover in the test cell demonstrated a linear conductivity response versus concentration, as also observed in pure solution up to saturation¹⁷; however, the signal strength and response slope were much lower than with a pure solution. Apparent slurry conductivity was only a fraction of the conductivity of pure liquid isolated from the same slurry. The discontinuous phases of liquid, solid, and gas formed a non-ideal electrical circuit between the electrodes, compared with pure liquid, and this situation explains the reduced conductivity readings. The signal strength ratio (apparent whole slurry conductivity / isolated liquid conductivity) at the bench under ambient conditions was very sensitive and varied between 0.01–0.20 over a small range (28–35%) of slurry total solids fraction with greater moisture content resulting in stronger signal. Due to difficulty of quantifying this factor *in vivo*, this effect was not compensated for. Instead, conductivity was interpreted directly as relative tracer concentration, as permitted by the fact that the reactor was operating at steady state and

apparent conductivity varies linearly with tracer concentration at constant temperature and slurry composition.

An Opto22 (Temecula, CA, USA) SNAP-PAC connected to a Gensym (Austin, TX, USA) G2 supervisory control and data acquisition system controlled the reactor and also recorded conductivity data once per second. Data was only valid at the end of each discharge airlock cycle, since measurement depended on sufficient process material in the bottom of the airlock to submerge the conductivity probes. Figure 4 graphically demonstrates this effect, where pseudo-square waves are produced as the electrical circuit is made and broken each cycle by the presence and discharge of process slurry. The baseline conductivity corresponds with the sulfuric acid and trace minerals dissolved in the slurry, and elevated values are associated with NaCl tracer present. Valve cycle time was set between 40–60 s to submerge the probes for a minimum of five seconds before discharge, and discharger valve positions were recorded to indicate data validity. At least three experimental replicates per residence time setting were required due to the widely spaced sampling intervals, but shorter intervals in different reactors may relax this requirement. As noted earlier, large-scale reactors may use continuous discharge valves and in that case, continuous conductivity measurement is possible and would likely reduce the need for replicates to generate sufficient data to reproduce the RTD.

Figure 4. Apparent conductivity signal at discharge airlock of steady state pretreatment reactor after NaCl tracer pulse. Pseudo-square wave nature indicated closing and opening of electrical circuit formed through slurry as discharge airlock chamber cyclically filled and emptied, while peak amplitude indicated apparent conductivity of the slurry.

Data were processed using numerical methods implemented by the authors in the Python 2.7 programming language. The conductivity data were parsed, taking the mean of the last three seconds prior to each airlock discharge cycle. Alternatively if continuous measurement is possible, this step could be omitted. The average baseline signal was subtracted, data were divided by the tracer dose to normalize each run (for equal reactor conditions), and 0.3 min was subtracted from run time to account for time spent in the tapered screw feeder that was not at reaction conditions. Next, a fitting curve was generated from the composite of all data points using a regularization-based signal smoothing method¹⁸, and subsequently, outliers were eliminated based on Chauvanet's criterion¹⁹ when they fell further than the computed cutoff value (typically ± 3 standard deviations) from the fitting curve. The Gamma distribution is a good

approximation for describing the RTD in reactor systems like this¹² and `scipy.optimize.curve_fit` was used to fit a gamma probability distribution to the valid data points.

$$E(t) = \frac{p_0}{\beta^\alpha \Gamma(\alpha)} (t - t_0)^{\alpha-1} e^{-(t-t_0)/\beta}, \quad (2)$$

where $E(t)$ is the RTD as a function of time and the remaining parameters were defined as follows.

$$\alpha = \frac{4}{s^2}, \quad (3)$$

$$\beta = \frac{1}{2} \sigma s, \text{ and} \quad (4)$$

$$t_0 = \tau - 2 \frac{\sigma}{s}, \quad (5)$$

where p_0 is a parameter used to scale the distribution to the data points, s is the skewness, σ is the standard deviation, τ is the mean residence time, and t_0 is the signal breakthrough time. The parameters of interest, τ , σ , s , and p_0 , were optimized and directly returned by the subroutine to best fit the data.

Unfortunately, due to the unknown amount of gaseous voids in the wet-granular slurry and difficulties obtaining a correction factor *in vivo* as mentioned earlier during bench study, it was not possible to connect $E(t)$ with salt concentration. Hence, it was also not possible to compare p_0 to the salt impulse and determine a mass balance. Nonetheless, $E(t)$ varies linearly with salt concentration and the RTDs are self-consistent for a single set of reactor conditions. All presented RTD curves (and experimental data points) are rescaled by p_0 so that the displayed p_0 is unity. Uncertainties were estimated using the standard deviation output of the

scipy.optimize.curve_fit function and two-standard deviation confidence intervals are reported here.

The Peclet number (Pe) indicates the degree of axial dispersion and was calculated by²⁰

$$Pe = \frac{8}{\sqrt{8\frac{\sigma^2}{\tau^2} + 1} - 1} \quad (6)$$

to enable direct numerical comparison between RTDs with differing means. A larger Pe value indicates less dispersion in the reactor, which is ideal.

An offline evaluation of the reactor RTD was made for one operating condition concurrently with online recording for validation of the tracer selection and to compare with the online method. One hundred grams of rutile titanium dioxide powder was premixed with the sodium chloride tracer doses before pulsing into the reactor. During online conductivity data collection, pretreated corn stover was hand-collected from the flash tank discharge with intervals equal to the valve cycling period of 45 s for this case. Samples were later analyzed for ash content²¹ (perturbed by total sodium chloride and titanium dioxide) and titanium dioxide concentration using a Foss (Hillerød, Denmark) XDS NIR spectrometer operated between 400–2,500 nm and calibrated with TiO₂-spiked pretreated corn stover. Data was processed similar to conductivity data with average baseline subtracted, data divided by tracer dose, outliers removed, and a Gamma distribution fitted to the data to yield the RTD function, $E(t)$, for both ash and NIR methods. This procedure was executed in triplicate.

RESULTS & DISCUSSION

The tracer validation trial indicated that soluble and insoluble compounds travel at the same rates at reaction conditions meaning no liquid/solid stratification was observed and that sodium chloride was an acceptable tracer for pretreating biomass. Titanium dioxide powder was used as a benchmark as it is insoluble and a good representative of the fines ($<100\ \mu\text{m}$) fraction of solids of the feedstock. We assumed the fines and larger particles within the slurry conveyed at equal rates as the moisture within the slurry tends to cohesively bind particles together. The results plotted in figure 5 indicate good agreement between ash- and NIR-based RTDs and indicate soluble NaCl traveled at the same rate as the insoluble TiO_2 . This validated NaCl as a suitable tracer for this system.

Figure 5. Online conductivity RTD measured over eight replicates with scaled data points plotted ($\cdot$). Outliers not used during signal processing are also shown ($\times$). Offline ash and NIR method RTDs are plotted for comparison. Similar results are obtained between the ash method (measuring NaCl & TiO_2) and NIR method (measuring only TiO_2), validating the use of soluble NaCl tracer. The flash tank section was included by the offline methods due to sampling location constraints and caused those RTDs to shift away from the online conductivity RTD.

Figure 5 presents the measured and reconstructed RTD using the online detection method. The online RTD shows some disagreement with the RTDs produced by the offline validation trial. The positions of the offline responses appear delayed and the distributions are also broader compared with the online conductivity method. Due to technical difficulties and personnel safety considerations, offline sampling directly from the reactor discharge airlock (where the conductivity probes were located) was not possible; instead, sampling occurred after the material had explosively flashed, traveled through the agitated flash tank, and dropped down a collection hose. The flash tank was not of interest with respect to the reaction zone RTD and likely contributed to the observed offline residence time delay and additional variance of the distributions. The disagreement between the offline methods and the online conductivity method indicates the importance of sampling location and favors the online method for accuracy.

In this type of reactor, process material is mechanically screw-conveyed either upwards or horizontally with no means to exit the reactor before the calculated plug flow time. However, a radial clearance gap between the auger screws and tubes to accommodate parallel anti-rotation stator bars may allow some fraction of material to slip backward around the flights and consequently exit the reactor at a later time. Therefore, the calculated residence time estimate, t_{th} , only signifies the expected start of the RTD with the distribution occurring after this time. As anticipated, the online electrical conductivity-based RTD presented in Figure 1 indicated a breakthrough response (t_0) at 11.6 min, a peak at 14.4 min, and a decay to baseline approximately 20 min after induction of NaCl tracer. The mean residence time of 15.3 ± 0.3 min was significantly longer than the calculated plug flow expectation of 10.7 min, which highlights

the importance of experimentally obtaining the RTD. The skew was $+0.97 \pm 0.36$ indicating a longer tail extending to the right as expected. The Peclet number, Pe , was 147 ± 13 , which is slightly higher than the experimentally obtained value of 106 in a previous study involving a twin-screw polymer extruder⁹. The back mixing of material around the horizontal tube auger flights is likely a major cause of RTD dispersion and shift of the mean time away from the estimate. Effects of transition sections, such as the large chute, and also flow irregularities within the upwards-conveying vertical impregnator also likely stratified movement of material, which delayed the initial response breakthrough, and shifted the mean time further away from the calculated time.

Different reactor operating conditions were also assessed during the development of this RTD measurement method and gave insight into the factors affecting residence time control. Both biomass feed rate and reactor tube auger speeds were varied to probe the effects of screw speed and tube fill fraction (Q/v). When feed rate was reduced to 326 kg/d while reactor tube speeds were adjusted to maintain the original theoretical time setting of 10.7 min (dashed red distribution in Fig. 6a), the breakthrough time of 12.2 min fell a relatively short delay past the calculated theoretical time. The mean residence time was 19.2 min, which is significantly longer than 15.3 min observed when the reactor was fed at the original 600 kg/d rate. Pe was reduced from 147 to 61, indicating more dispersion with reduced throughput as can clearly be seen by the difference in distributions.

Figure 6. Comparison of RTDs for different operating conditions (a). The same RTDs are time-normalized (dimensionless) for direct graphical comparison of relative dispersion (b).

Further, reactor tube 1 and 2 auger speeds were increased to a maximum proportionally, so as to keep their volumetric fill fractions equal, while maintaining 600 kg/d dry biomass feed rate. This significantly shortened the breakthrough time to 6.2 min as demonstrated by the dashed blue distribution in Fig 6a, which again was just past the theoretical time of 5.8 min indicating the validity of equation 1. However, the mean time of 9.2 min was proportionally much further delayed from the theoretically expected time, as noted by the low value of Pe at 45, even though the original feed rate was maintained. Figure 6b presents dimensionless RTDs after normalizing by each distribution's mean, where the shape graphically demonstrates relative dispersion differences directly between conditions. The common operating variable between these two additional cases with diminished Pe is the reduction in fill fraction from 0.44 to 0.20 and the

reduction in fill fraction likely increases back-mixing by exposing a larger fraction of each screw pocket to the auger edge where material can slip past the flight to the next pocket.

Based on experimental observations, the following predictive model was developed in which estimated mean residence time (τ) is computed from the theoretical residence time (t_{th}) found using equation 1 and the reactor tube fill fraction (Q/v):

$$\tau = t_{th} \left(k_0 + k_1 \frac{v}{Q} \right), \quad (7)$$

where k_0 represents the non-ideality causing delay between theoretical time (t_{th}) and the actual breakthrough time (t_0), and k_1 is a scaling term to account for the delay between the breakthrough time and mean presumed to be caused by reduced fill factor and subsequent material slippage backwards around auger flights. This form is similar to the model presented by *Nachenius et. al.*, although it is interesting to note they have associated delay time with *increased* fill fraction (Q/v), which is the opposite of our hypothesis and observations. They reported a wider range of fill fractions than used in this study, and it is likely that above a certain limit, material crests and falls over the screw shaft to the next pocket rather easily compared to when operated under our reactor's manufacturer-recommended limit of $Q/v \leq 0.60$. This phenomenon would actually *increase* dispersion above a certain fill fraction, and could offer an explanation for the discrepancy. Additionally, that study also used a step change halting all inlet flow during screw operation, which could indirectly affect the fill fraction by operating at non-steady state flow and confound the measured degree of dispersion. k_0 and k_1 were determined by regression with experimental data as 1.165 ± 0.196 and 0.117 ± 0.049 , respectively, and Figure 7 demonstrates good agreement between the predicted and experimental mean residence times.

Figure 7. Parity plot for model and experimental mean residence times.

This online conductivity method offers major time and cost advantages compared with offline approaches. Six additional technicians were required during the fast-paced physical sample collection of the offline validation method. Two technicians are typically required to operate the pretreatment reactor and no additional labor was required to apply the online RTD measurement method. Recorded conductivity data were immediately available for processing after the experiment. In contrast, substantial additional analytical labor and time is needed for ash or NIR determination. Conductivity instrumentation and probes cost 2,400 USD (2014), excluding the data logging equipment.

CONCLUSION

An online electrical conductivity method for obtaining residence time data from continuous biomass pretreatment reactors was successfully developed using sodium chloride tracer and a

predictive model for the mean residence time was developed. The mean residence time significantly differed from the theoretical value and accentuates the importance of experimentally measuring RTD instead of relying on theoretical calculations. This low-cost and rapid technique enables further exploration of process factors affecting pretreatment RTD, allows routine residence time verification, and is also applicable to other processes that involve wet-granular slurries, such as wood pulping and food processing.

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