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TEXTILE TECHNOLOGY

Mitigation of Whitefly Honeydew Levels on Cotton Lint via Thermal and Citric Acid Treatment

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INTERPRETIVE SUMMARY

Cotton contaminated by insect honeydew, termed sticky cotton, is a major concern to the textile industry. There are currently no methods to remediate the contaminated cotton before arrival at the textile mill, and if not carefully handled it may result in machine shutdown. A method for handling sticky cotton prior to processing is described in this work that involves the addition of a chemical catalyst to the contaminated cotton and subsequent heat treatment. Results show a decrease in honeydew sugar levels, indicating that the method may be used to eliminate stickiness on contaminated cotton so that processing of the cotton will not lead to mill shutdown.

ABSTRACT

Contamination of cotton lint by insect honeydew can interfere with carding, roving, and spinning processes at the mill, and thus presents a major concern for the textile industry. Several methods exist for the detection of insect honeydew on cotton lint, but there are currently no methods available for the remediation of insect honeydew contamination. The objective of this study was to determine the effectiveness of a heat-activated catalyst in decreasing the honeydew level of a contaminated cotton sample. Cotton lint heavily contaminated with whitefly honeydew was treated with citric acid and subsequently subjected to elevated temperatures for varying periods of time. Sugar concentrations on the cotton lint were analyzed as a function of temperature and heat duration using high-performance anion-exchange chromatography. Levels of honeydew sugars decreased for catalyst-treated cotton compared with untreated cotton when heated at a temperature of 160°C. Chemical treatment of contaminated cotton in conjunction with elevated temperatures may be the basis of a potential method for the mitigation of stickiness on cotton prior to processing at the mill.

Sugar contamination on cotton lint that causes fibers to stick to processing equipment and leads to machine shutdown is a major concern of the textile industry. Though some cases of lint stickiness are due to plant physiological sugars arising from fiber immaturity, it is generally agreed that insect honeydew arising from whitefly (*Bemesia argentifolii*) or aphid (*Aphis gossypii*) infestation is responsible for 80-90% of all cases of lint stickiness (Sisman and Schenk 1984). A wide range of methods exist for the detection of sugar contamination on cotton (Brushwood and Perkins 1993), and though none of these methods possesses all of the characteristics required to successfully screen all cotton bales reliably and cost effectively, many are currently in use to identify sticky cotton. Once cotton has been identified as being sticky there are few reliable and cost effective methods available for remediation. The most widely utilized method involves blending sticky bales with non-sticky bales in the laydown in a ratio of ~ 1:10. Though blending can be successful when a small number of bales have been identified as being sticky, problems arise when the number of sticky bales exceeds what can be properly blended. It is therefore desirable to not only identify cotton as being potentially sticky, but to treat it in order to mitigate the stickiness before arrival at the laydown.

One approach to reducing the amount of sugar on the surface of cotton lint involves promoting microbiological activity on the fibers (Heuer and Plaut 1985). Under suitable conditions of temperature and moisture, fungal species such as *Aspergillus niger* will grow naturally on honeydew-contaminated cotton, consuming the sugars present on the surface of the fiber. A deleterious side effect of this growth is concomitant degradation of fiber components, in-

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cluding cellulose and pectin, which leads to an unacceptable reduction in fiber quality.

A second approach involves the utilization of enzymes such as α-glucosidase to promote hydrolysis of honeydew oligosaccharides to constituent glucose and fructose monomers (Henneberry et al. 1997). This method requires cotton moisture of ~9% or higher for enzyme activity to have a significant effect, and the glucose and fructose resulting from enzymatic hydrolysis can themselves promote stickiness. Subsequent reduction of glucose and fructose levels currently depends upon microbial activity and its associated detrimental effects.

Previous research has demonstrated that trehalulose and melezitose, the primary oligosaccharides comprising whitefly honeydew, are subject to thermochemical degradation at sufficiently high temperatures (Gamble 2002). At 200°C, trehalulose degrades relatively quickly, but melezitose degrades much more slowly. Addition of a small amount of organic acid catalyst to pure melezitose was shown to greatly increase the rate of thermochemical degradation. In the present study, an organic acid catalyst is applied to the surface of cotton contaminated with whitefly honeydew in order to determine its effects on degradation *in situ* and to determine optimal conditions of temperature, heat duration, and catalyst concentration under which degradation of honeydew will occur with minimal effect to fiber quality. Ideally, degradation should occur quickly at temperatures sufficiently low to avoid erosion of cotton fiber quality parameters such as length and strength, and the catalyst used should be cost efficient and easy to apply.

MATERIALS AND METHODS

A 10-g sample of cotton heavily contaminated with whitefly honeydew was obtained from the USDA-ARS Western Cotton Research Laboratory in Phoenix AZ. The sample was not previously subjected to elevated temperatures. The cotton was conditioned at 72°F and 65% relative humidity prior to treatment. A portion of this cotton was subdivided into 0.10-g samples that were formed into ~4 in^2 webs for subsequent heat treatment without the catalyst. A second portion of contaminated cotton, identified as Treatment 1, was subdivided into 0.10-g samples that were formed into ~4 in^2 webs. The webs were then placed into a sealed 500 ml container along with an excess of citric acid (Sigma-Aldrich Co., St. Louis MO) and shaken until the weight of the resultant web was constant. Average weight gain due to incorporation of citric acid was 12%. A third portion of the contaminated cotton, identified as Treatment 2, was saturated with anhydrous citric acid powder by thorough mixing the cotton lint in an excess of citric acid powder. Mixing was achieved by repeated opening of the lint ball, dusting the outer surfaces with citric acid, and folding. The resultant sample, 50% citric acid by weight, was subsequently subdivided into 0.20-g samples that were formed into ~4 in^2 webs for heat treatment. A series of webs for each treatment were heated at either 160°C or 200°C for 0, 1, 2, 5, 10 or 20 minutes in a forced draft oven.

Heated webs were then extracted in 10.0 ml deionized water. An aliquot of 0.5 ml of extract was analyzed for individual sugars using high-performance anion-exchange chromatography (HPAEC) performed on a Dionex DX-500 (Dionex Corp., Sunnyvale CA) using pulsed amperometric detection. Two Dionex Carbopac PA-1 (4 x 250 mm) columns were connected in series and elution was carried out at 0.75 ml/min using 200 mM NaOH as the mobile phase and a sigmoidal gradient of 0 to 500 mM NaOAc.

Concentrations of melezitose and trehalulose as a function of heating time on treated and untreated samples are described by

$$[C]=[C]_0 e^{-k(T)t} \quad \text{[Eq. 1]}$$

where t is the heating time, $[C]_o$ is the concentration of either trehalulose or melezitose at t = 0 minutes, [C] is the concentration of trehalulose or melezitose at time t, k is the (temperature dependent) rate constant, and T is the temperature. Values of the rate constant k were determined by non-linear regression on Equation 1 using experimentally determined values of time and concentration. All regressions and analyses of standard error were performed using SigmaPlot 5.0 (SPSS Science, Chicago, IL).

Samples of uncontaminated, moderately contaminated, and severely contaminated cotton were also treated with citric acid in order to determine the efficacy of citric acid treatment on mechanical stickiness ratings. Mechanical stickiness ratings were obtained using the Sticky Cotton Thermodetector (IRCT, Montpellier, France). Operation of the Sticky Cotton Thermodetector was as described previously (Brushwood and Perkins, 1993). Two 1.0-g portions each of an uncontaminated cotton, a moderately contaminated cotton, and a severely contaminated cotton were formed into ~60 in^2 webs. The webs were placed into a sealed 2-gallon container along with an excess of citric acid and shaken until the weight of the resultant web was constant. Average weight gain due to incorporation of citric acid was 12%. The resultant citric acid-treated webs were heated at 160°C for either 0 min or 15 min for each level of contamination. Following heat treatment, the webs were tested for stickiness by the Sticky Cotton Thermodetector. Rating categories developed previously in which 0 indicates no contamination, 1 indicates mild contamination, 2 indicates moderate contamination, 3 indicates heavy contamination, and 4 indicates severe contamination were used to determine the effect of the different treatments on cotton stickiness (Perkins and Brushwood 1995).

RESULTS AND DISCUSSION

Cotton contaminated with whitefly honeydew exhibits high levels of both melezitose and trehalulose. When the untreated cotton is heated to160°C or 200°C, both saccharides decrease in concentration due to thermochemical reactions. Figures 1 and 2 show the concentration of trehalulose and melezitose, respectively, on untreated cotton lint as a function of temperature and heat duration. The concentration of trehalulose decreases by 11% when untreated cotton is heated at 160°C for one minute. Heating the cotton at 200°C for one minute decreases the initial trehalulose level by 66%. The rates of degradation of melezitose on untreated cotton at 160°C and 200°C are much slower than those of trehalulose. Heating the untreated cotton for one minute at 160°C results in a 2% decrease in melezitose concentration, while one minute at 200°C results in a 17% decrease. The rates of degradation for trehalulose and melezitose on cotton without citric acid treatment are described by Equation 1, and resulting values of the rate constant k are shown in Figures 3 and 4, respectively.

These results indicate that subjecting untreated cotton contaminated with honeydew to elevated temperatures results in decreased levels of the primary honeydew sugars trehalulose and melezitose. The duration of heat required to achieve a substantial degree of honeydew mitigation, however, is unrealistically long for practical utilization as a remediation method. Heating sticky cotton at 200°C would require a residence time of 12 minutes in order to decrease melezitose levels by 90%. It is generally agreed that temperatures in excess of 180°C may lead to irreversible and detrimental changes to fiber quality after a relatively short heating interval (Anthony 1994). Heating untreated contaminated cotton at 160°C could potentially decrease the risk of incurring fiber damage, but the time required to decrease the melezitose level by 90% would be 2 hours. Aside from being unrealistic economically, the effect of 160°C heat for this length of time would almost certainly lead to irreversible fiber damage. Heating contaminated cotton at temperatures less than 160°C for longer lengths of time is not a viable option due to the fact that the thermochemical degradation reactions occur only at temperatures in excess of the individual melting points of the sugars and the or-

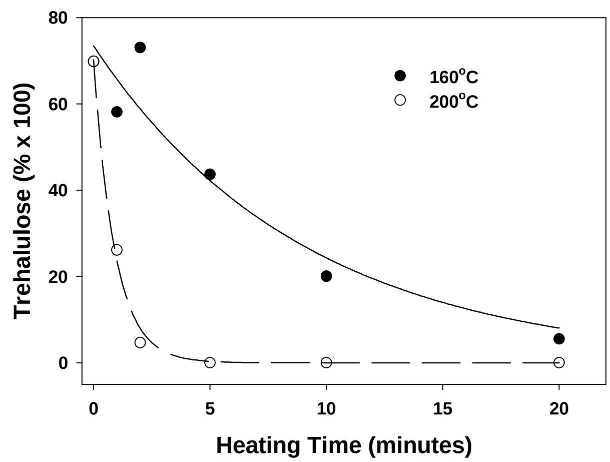

Figure 1. Concentration of trehalulose on cotton lint contaminated with whitefly honeydew heated at 160°C and 200°C for 1, 2, 5, 10, and 20 minutes.

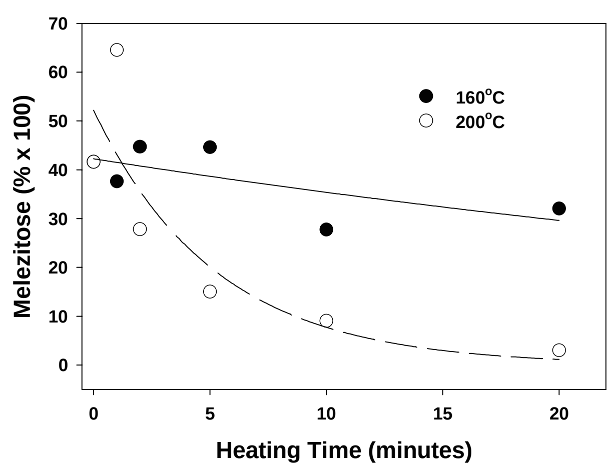

Figure 2. Concentration of melezitose on cotton lint contaminated with whitefly honeydew heated at 160°C and 200°C for 1, 2, 5, 10, and 20 minutes.

ganic acid catalyst. Many sugars, with the notable exceptions of trehalulose and fructose, melt at temperatures greater than 150°C. Trehalulose melts at approximately 100°C, and partial mitigation of whitefly honeydew contamination may be possible at this temperature. As a general method for the remediation of aphid honeydew contamination, however, this temperature is unsuitable since trehalulose is a relatively minor constituent of aphid honeydew (Hendrix 1999).

In order for heat treatment to be a viable method for honeydew mitigation, it is necessary to find a means of increasing the rate of thermochemical degradation at a given temperature. Previous work has shown that the rate of thermochemical degradation of saccharides can be substantially increased by the introduction of an organic acid catalyst (Gamble 2002). When contaminated cotton is treated with 50% (w/w) citric acid powder (Treatment 2) and subsequently subjected to elevated temperatures, the rate of thermochemical degradation of both trehalulose and melezitose increase dramatically, as indicated by the rate constants (Figures 3 and 4, respectively). Treatment reduces the concentration of trehalulose by 74% after one minute at 160°C (Figure 5). The effect of Treatment 2 is even more dramatic on melezitose, whose concentration decreases by 75% when heated at 160°C for one minute. Heating at 200°C for one minute decreases trehalulose concentration by 90% and melezitose by 88%.

The results from Treatment 2 are encouraging from the standpoint of honeydew remediation, but the application of this treatment on a large scale is impractical because the excessive amount of citric acid makes it cost prohibitive, as well as affecting the quality of the treated cotton lint. It is therefore desirable to treat the honeydew contaminated cotton with citric acid in such a way as to decrease the quantity needed while retaining the positive degradative effects. When cotton lint contaminated with honeydew is treated with 12% (w/w) citric acid powder (Treatment 1) and subjected to elevated temperatures, the rates of thermochemical degradation of trehalulose and melezitose are increased substantially over those of the untreated samples, although they are substantially slower than the samples treated with 50% (w/w) citric acid (Treatment 2) (Figures 3 and 4). Nevertheless, the concentration of trehalulose decreases by 80% and melezitose by 75% for cotton treated with 12% citric acid (Treatment 1) and heated at 160°C for four minutes.

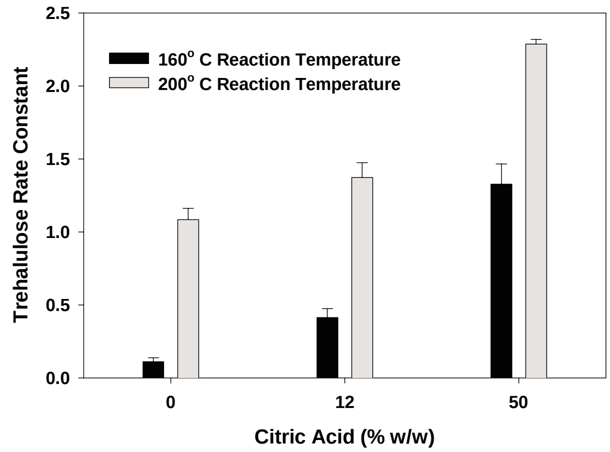

Figure 3. Effect of citric acid treatment and temperature on the rate constant for the thermochemical degradation of trehalulose of cotton lint contaminated with whitefly honeydew. Bars indicate standard error.

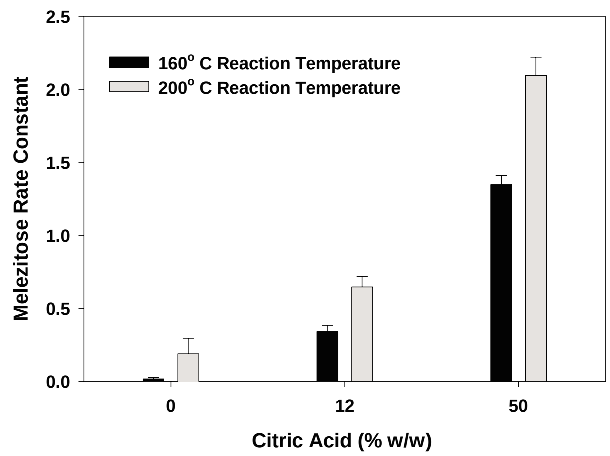

Figure 4. Effect of citric acid treatment and temperature on the rate constant for the thermochemical degradation of melezitose on cotton lint contaminated with whitefly honeydew. Bars indicate standard error.

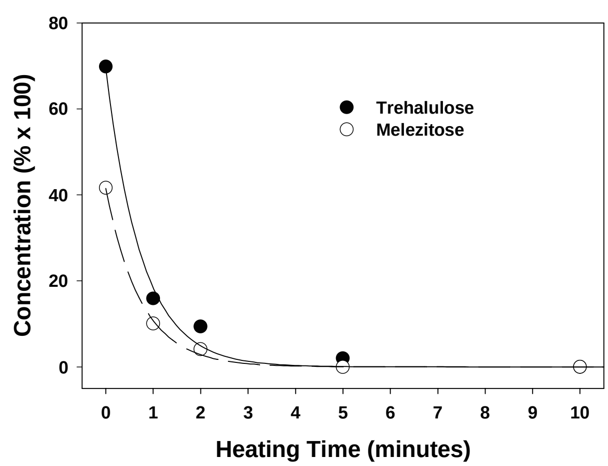

Figure 5. Concentration of trehalulose and melezitose on cotton lint contaminated with whitefly honeydew treated with 50% (w/w) citric acid and heated at 160°C for 1, 2, 5 and 10 minutes.

Through the application of citric acid powder to the surface of cotton lint contaminated with honeydew, the effective temperature and duration of heating required to substantially thermochemically degrade honeydew sugars are decreased. Contaminated cotton thus treated can be heated at 160°C for several minutes to realize a substantial decrease in surface sugar contamination. These temperatures and durations of heating are also of a magnitude low enough to potentially avoid significant concomitant damage to the cotton fibers. This method is consequently being studied as a possible means of stickiness mitigation.

The addition of 12% (w/w) citric acid to contaminated cotton (Treatment 1) and heating at 160°C for 15 minutes reduces the stickiness rating by 50%, based on the Sticky Cotton Thermodetector. Moderately contaminated cotton with an initial stickiness rating of 2 was reduced to a stickiness rating of 1, while severely contaminated cotton was reduced from 4 to 2 in stickiness rating. Residual stickiness is due apparently to decomposition products such as caramel polymers. The citric acid used as catalyst does not contribute to stickiness, as evidenced by the fact that the non-sticky sample treated with citric acid remained non-sticky after heat treatment. A potentially adverse effect of the method is formation of brown reaction products (i.e. caramel) that negatively affect the HVI color classification measurement, thus leading to discounts of the treated cotton, though the color change caused by citric acid and heat treatment is a surface effect and removed during scouring and bleaching.

Other challenges remain to be overcome if this method is to have utility for the cotton industry. First, since the method requires heating of the cotton on a large scale, the gin is the most obvious place for this treatment to be applied. This consequently requires that cotton be identified as being potentially sticky before arrival at the heating component of the gin. Second, the amount of citric acid needed to catalyze the degradation of sugars is theoretically much smaller than that used in the present work. Previous work indicated that approximately 10% (w/w) citric acid relative to the amount of sugar present is sufficient for effective catalysis (Gamble 2002). Since honeydew sugars may constitute only 1% of the weight of a very sticky cotton sample, the theoretical amount of citric acid needed would be very small; 0.1% (w/w) relative to the cotton sample. The large difference between the theoretical amount of citric acid catalyst needed and the amount actually used in the current work arises as a consequence of the way in which the catalyst is delivered to the cotton sample and mixed. A more effective method of catalyst application may involve light dusting of sticky cotton at picking, where the honeydew contamination is nearly entirely at the surface of the open boll. This and other techniques to optimize catalyst application to contaminated cotton lint for heat treatment at the gin are currently under investigation.

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