Comparison of the Moisture Adsorption Properties of Starch Particles and Flax Fiber Coatings for Energy Wheel Applications

Wahab O. Alabi, Abdalla H. Karoyo, Easwaran N. Krishnan, Leila Dehabadi, Lee D. Wilson,* and Carey J. Simonson

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ABSTRACT: The adsorption−desorption behavior of flax fibers (FFs) is reported in this paper. FFs are a potential desiccant material for air-to-air energy wheels, which transfer heat and moisture in building heating, ventilation, and air conditioning (HVAC) systems. The raw FFs sample was subjected to physical modification, followed by complementary material characterization to understand the relationship between its structure and its moisture uptake performance. The surface and textural properties of the modified FFs were determined by gas adsorption (N₂, H₂O) and gravimetric liquid water swelling studies and further supported by spectroscopic (infrared and scanning electron microscopy) results. A FF-coated small-scale energy exchanger was used to determine the moisture transfer (or latent effectiveness; ε₁) using single-step and cyclic testing. The FF-coated exchanger had ε₁ values of ~10 and 40% greater compared to similar exchangers coated with starch particles (SPs) and silica gel (SG) reported in a previous study. The enhanced surface and textural properties, along with the complex compositional structure of FFs and its greater propensity to swell in water, account for the improved performance over SPs. Thus, FFs offer an alternative low-cost, environment-friendly, and sustainable biodesiccant for air-to-air energy wheel applications in buildings. The current study contributes to an improved understanding of the structure−function relationship of biodesiccants for such energy wheel applications.

1. INTRODUCTION

The heating, ventilation, and air conditioning (HVAC) system design is based on the principles of thermodynamics, fluid mechanics, and heat transfer technology. The goal is to provide thermal comfort and acceptable indoor air quality through efficient heat and mass transfer processes. HVAC systems are an essential technology in various types of buildings (residential, office, hotels, hospitals, etc.) where safe healthy conditions are maintained by regulating the temperature and humidity between indoor and outdoor air supplies.¹ The current estimates (30−50%) of energy consumption and greenhouse gas emissions go towards heating and cooling of commercial, residential, and industrial buildings in Canada¹ and the EU.² The rate of energy consumption has been projected to increase exponentially over the next century because of climate change and increasing energy needs of some developing countries.³ Because an associated increase in cost concurs with greater energy demands, there is a dire need to develop energy- and cost-efficient HVAC systems.

The incorporation of air-to-air energy exchangers (AAEEs) into HVAC systems has been reported to alleviate the difficulties associated with enormous energy intake of conventional systems.⁴,⁵ An important attribute of AAEEs is that the energy wheel employs a rotating metallic substrate coated with a desiccant, enabling recovery of moisture and heat from air streams during exchange between indoor and outdoor air supplies. This type of HVAC system is the most common and constitutes more than 75% of the market demand.⁶ One parameter used to determine the performance of energy wheels is the latent effectiveness (ε₁) that is defined as the ratio of the actual rate of moisture transfer between the supply and exhaust air streams to the maximum possible rate of transfer between them.⁷ Extensive research over the past decades has focused on modeling the heat and mass transfer of desiccant-coated energy wheels, along with their performances.⁸−¹⁵ Several empirical correlations have been proposed to estimate the effectiveness of wheels with a certain geometry and material composition at

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certain operating conditions.\textsuperscript{13,16} Results showed that $\varepsilon_l$ can vary depending on the angular speed, air flow rate, and the interaction between the coating material and the metal substrate of the heat exchanger. Moreover, the sorption properties of desiccants play an important role in the moisture recovery of energy wheels.\textsuperscript{17} Thus, desiccants with good sorption capacity and low activation barrier for moisture uptake are preferred materials for the energy wheel surface coating.\textsuperscript{17} Also, sustainability of the desiccant is gaining increasing importance for energy wheel applications.

The use of mineral-based desiccants, such as silica gels (SGs),\textsuperscript{18-21} activated alumina,\textsuperscript{22-24} zeolites,\textsuperscript{21,25} metal organic frameworks,\textsuperscript{25-28} and hybrid substrates,\textsuperscript{29-31} has been reported in energy wheels. Interest in the research and utilization of agricultural biomass is underway because of the notable water adsorption–desorption capabilities, high abundance, low cost, and material sustainability.\textsuperscript{32} Previous research has shown that appreciable differences exist between the performance of conventional SGs and high amylose starch (HAS), where greater sorption rate and uptake capacity were reported for HAS.\textsuperscript{33,34} More recently, starch particles (SPs) were shown to possess greater moisture recovery, approximately $\sim$3- and 6-fold greater over HAS and SG, respectively.\textsuperscript{6} The unique granule structure of SPs, along with its conformational motility and accessibility of its abundant hydrophilic surface $\sim$OH groups represent some of the key properties of a good desiccant material.\textsuperscript{6} Although the adsorption–desorption properties of SPs under dynamic flow conditions are favorable, the relative longevity, handling, and disposal cost are technical issues of some concern for large-scale applications.\textsuperscript{15,36} Thus, other biomass materials with improved mechanical/thermal stability and notable microbial resistance such as flax fiber (FF), wheat, and cassava are stimulating research interest as potential alternative desiccants for moisture uptake in energy wheel systems.

This study extends existing research efforts on FF by demonstrating its utility as a biomass desiccant with improved sorption properties and regeneration capability with evidence of stability over multiple adsorption–desorption cycles. Herein, the biodesiccant performance of FFs was compared against SPs, where the latter was originally reported elsewhere.\textsuperscript{6,37} The surface and textural properties of the FFs were characterized using complementary spectroscopic and thermoanalytical techniques, while the adsorption–desorption properties and performance ($\varepsilon_l$) were studied using a small-scale energy exchanger. Overall, this comparative study of two biomass desiccants reveals that FFs possess good sorption capacity relative to SPs, where the structure–adsorption properties of the FFs were related to its performance at typical conditions relevant to AAEEs.

2. RESULTS AND DISCUSSION

2.1. Material Characterization Results. The characterization results of the FFs material are presented herein along with the results of SPs that were originally reported\textsuperscript{6,37} as part of a wider study.

2.1.1. Fourier-Transform Infrared Spectral and Particle Size Distribution and Gas Adsorption Results. The IR spectra, PSD, and nitrogen adsorption results for the FFs and SPs solids are presented in Figure 1. In Figure 1a, the Fourier-transform infrared (FT-IR) spectra of the materials show several bands that are assigned to different vibrational signatures. The strong bands at $\sim$1081–1150 cm$^{-1}$ (C=O–H bending and C–O stretching), 2970 cm$^{-1}$ (C–H stretching), and 3390 cm$^{-1}$ (O–H stretching) were assigned, in line with a previous report.\textsuperscript{38-40} The medium intensity band at $\sim$1640 cm$^{-1}$ was assigned to C–O bending associated with the OH group that relates to adsorbed water in the amorphous regions of biomass such as flax and starch.\textsuperscript{38-40} This band has greater intensity for the FFs material (cf. Figure 1a).

Figure 1. (a) FT-IR spectra, (b) PSD, and (c,d) N2 gas adsorption isotherms at 77 K of SPs and FFs.
In addition to the 1640 cm\(^{-1}\) band, other unique bands are observed for the FFs at the 900–1730 cm\(^{-1}\) region (denoted by asterisks) that are assigned to the vibrational bands of the cellulose backbone and other polysaccharide components (e.g., hemicellulose, cellulose, pectins, and lignins).\(^{41,42}\) Several unique vibrations in Figure 1a may be diagnostic of hemicellulose and pectin fractions of the FFs, such as C–O–C symmetric glycosidic stretching at ca. 900 cm\(^{-1}\), CH\(_2\) bending of cellulose ca. 1430 cm\(^{-1}\), and the C–O ring stretching/C–O–H bending vibration of the cellulose backbone ca. 1050–1150 cm\(^{-1}\).\(^{43}\) In particular, the region ca. 1500 cm\(^{-1}\) is diagnostic for lignins\(^{44,45}\) and is prominent in the spectrum of FFs (cf. Figure 1a). The band at 1735 cm\(^{-1}\) is unique for the FFs and reveals the presence of pectin biopolymers in this fiber sample.\(^{43}\) In Figure 1b, the particle size distribution (PSD) analysis shows the volume distribution of the samples with respect to the particle size, where the average particle diameter (dp) for the SPs is ~15 μm (cf. Table 1).

Table 1. Physicochemical Properties of the Biomass Desiccants\(^{6,57}\)

| materials | particle diameter (μm) | average pore width (PW) (Å) | PV (cm\(^3\)/g) | accessible SA (m\(^2\)/g) | water swelling (%) |
|-----------|------------------------|-----------------------------|-----------------|---------------------------|-------------------|
| FFs       | >100                   | 23.1                        | 0.000036        | 0.229                     | 2015              |
| SPs       | 1.5                    | 80.6                        | 0.0065          | 2.890                     | 452               |

The N\(_2\) adsorption isotherm profiles of the FFs and SPs samples are presented in Figure 1c,d, where the textural properties are summarized in Table 1. The isotherm profiles of the biodesiccant materials in Figure 1c,d are consistent with type II adsorption isotherms that are characteristic of nonporous/microporous solids, according to the Brunauer, Emmett, and Teller (BET) theory.\(^{46}\) In general, natural flax is known to have a very small surface area (SA) and pore volume (PV) structure, as compared to most biomaterials, which are in good agreement with the results in Table 1. According to the N\(_2\) adsorption isotherms (cf. Figure 1c,d), uptake by FFs (b) occurs at higher relative pressures (P/P\(_0\) ≈ 1) and may be related to the limited SA and porosity of this material. It can be inferred from the above results that the diffusion of N\(_2\) molecules is the rate-limiting process in the FFs sample. In contrast, SPs (a) show greater uptake at all relative pressures with saturation at P/P\(_0\) ≈ 1. The small differences in N\(_2\) adsorption by the two materials is understood in terms of the textural and SA properties where SPs have greater SA over the FFs. Notwithstanding the limitations of the BJH model for estimating the textural properties of such biomass, the negligible PV in Table 1 for these materials is further evidenced by the negligible hysteresis loops (cf. Figure 1c,d), especially for FFs. The SA and PV estimates derived from gas adsorption herein for FFs and SPs show similar behavior to that observed in cellulose obtained from cotton linters,\(^{33}\) further illustrating the nonporous nature of the biomass reported herein.

2.1.2. Scanning Electron Microscopy Imaging Results. Figure 2 shows the scanning electron microscopy (SEM) micrographs for SPs (a) and FFs (b) samples. The images of SPs in Figure 2a are characterized by spherical particles with smooth edges and a variable size range because of particle–particle aggregation, as described in the PSD results given above (Figure 1b). The granular size of SPs was previously estimated to be <1.5 μm (cf. Table 1), but larger granules are evident in the SEM results. Larger particles (>1.5 μm) are likely due to aggregation of fine granules of SPs, in agreement with the bimodal distribution of SPs, according to particle sizes ≥100 μm in Figure 1b.

Unlike SPs which possess spherical morphology, the FFs are made up of long fibrils with variable particle sizes (>100 μm). The greater particle size and fibrous nature of FFs impart specific features with unique textural properties because of the presence of void volume in the structure that varies depending on the particle density. The structure of flax is made up of cellulose micro fibrils bound together into larger fiber bundles of a hemicellulose–pectin–lignin matrix. The SEM images reveal that the FFs have a more complex surface structure when compared against the SPs that possess a well-defined spherical morphology. The structural complexity of FFs may relate to the presence of fats, waxes, lignins, hemicellulose, and pectin fractions,\(^{48}\) consistent with the FT-IR results reported above.

2.1.3. Gravimetric Swelling Test Results. Unlike the use of molecular nitrogen as a probe for gas adsorption studies, water vapor uptake by biopolymers provides complementary information about the surface and morphological material properties. The difference between nitrogen and water vapor relates to greater polarity and smaller molar volume of water relative to nitrogen. Greater water swelling was reported for SPs that was attributed to the flexible and hydrophilic nature of this biopolymer, along with its propensity to undergo significant volume changes upon uptake of water due to swelling.\(^{6,49,50}\) The granular structure of SPs and the abundant polar functional (–OH) groups that originate from amyllose (AM) and amylpectin (AP) fractions provide sites that can adsorb water via hydrogen bonding. Similar to the SPs, FFs can undergo H-bonding via many of its polar groups because of its hygroscopic nature, in agreement with the water swelling results in Table 1. The greater swelling properties of FFs compared to SPs in water are accounted for by the hydrophilic nature of FFs, in agreement with the multicomponent biopolymer composition of FFs, and its amorphous fibril structure. Natural plant-based fibers are semicrystalline in nature because of an amorphous fraction that contains lignins,
pectins, and (hemi)cellulose biopolymers, and a crystalline cellulose fraction. The major components that contribute to water uptake are cellulose, hemicellulose, pectins, and lignins. According to literature reports, hemicellulose constitutes a major part of the amorphous fraction of plant fibers, where it is known to play a key role in moisture storage. The pectin fraction is composed of highly polar carboxylate anion groups at ambient pH conditions that are known to interact strongly with water. Also, the porosity and surface properties of FFs are influenced by the particle size and pore domains, where water can occupy the void sites of the material upon exposure to a humid environment. Thus, these factors account for the complex structure of FFs and the unique water uptake and swelling ability of FFs versus SPs.

2.2. Moisture Adsorption−Desorption Performance Test. An FF-coated small-scale exchanger was developed to study its moisture adsorption−desorption performance, as shown in Figure 3. The exchanger is exposed to a step change in humidity (known as single step change experiments) and a series of step changes in humidity (known as cyclic tests), where the results were compared to those previously reported for the SP-coated exchanger. The details related to the preparation process of the FF-coated exchanger and the operation of the test facility are outlined further in Section 5.

2.2.1. Single-Step Testing. The response of the FF-coated small-scale exchanger in terms of the variable and normalized humidity (W) is presented in Figure 4. The normalized humidity ratio (W) is defined as the ratio of the difference in the exchanger outlet humidity at any time (t) during the experiment to its maximum difference during the step test, which is mathematically defined using eqs 1 and 2.

\[ W(t) = \frac{W_{out,t} - W_{out,t=0}}{W_{out,final} - W_{out,t=0}} \]  

(1)

\[ W = \frac{kg_{water vapor}}{kg_{dry air}} \]  

(2)

W is the humidity ratio of the air stream at the exchanger outlets. The humidity ratio of the air streams at the inlet and outlet of the exchanger are calculated from the temperature and relative humidity measurements using the established ASHRAE standard method. During the adsorption process (cf. Figure 4a), there is a gradual increase in the value of W with time, where it attains a value of 0.9 within the initial few minutes. This shows that the FF-coated exchanger attained 90% of its equilibrium moisture content during the initial 180 s period. Near to 450 s, the humidity level of the supply and exhaust air streams reached a comparable moisture content, where W = 1.

In contrast, the constant W during the adsorption process for SPs (W = 0) in the initial few minutes indicates that it adsorbs all of the moisture during the initial stages (cf. Figure 4c), where no net change in the normalized humidity results upon saturation of the adsorption sites. During the desorption process for the FFs (Figure 4b), an opposite trend is observed with respect to the variation of W with time. Within the first 150 s, the value of W decreased from 1 to 0.2, where ca. 400 s was required for W to reach a zero value.

Figure 3. Photograph of the FF-coated small-scale energy exchanger. Photograph courtesy of Wahab Alabi. Copyright 2020.

Figure 4. Normalized humidity response of the FF-coated small-scale exchanger during (a) adsorption, and (b) desorption. SP- and FF-coated small-scale exchanger for a single step change at 23 °C during (c) adsorption and (d) desorption.
It is noteworthy that the desorption process of moisture by the FFs occurs more rapidly relative to the SP-coated exchanger, as shown by the more rapid decay for the humidity response profile in Figure 4d. The observed differences relate to the surface chemistry and textural properties of FFs, as supported by the unique gravimetric water swelling results. The foregoing indicates that the kinetics and modality of adsorption—desorption processes vary for the FF- and SP-coated exchanger, in accordance with the offset in their physicochemical properties described above.

2.2.2. Cyclic Test Results. The normalized humidity profiles for the adsorption and desorption process during cyclic tests are shown in Figure 5. The profiles were obtained after more than 1 h of operation when the system had reached a quasi-steady state. During the adsorption process, there is transfer of moisture from the humid air stream to the desiccant surface sites as the moist air passes through the exchanger. A reverse effect is observed during desorption as the desiccant transfers the adsorbed water to the dry air stream, resulting in a more humid air stream. This adsorption—desorption process continues throughout the cyclic operation test. The value of $W$ varied from 0 to 1 during the cyclic operation, which is similar to the result of the transient operation. More importantly, the rate of moisture uptake and removal during the test was fairly constant throughout the recorded cycles and provides support that the sorption process is at equilibrium, and all sorption sites retain their structural integrity, according to the stability observed throughout the cyclic experimental test evaluation.

2.2.3. Latent Effectiveness of the Exchanger for the Single Step and Cyclic Tests. The performance of a desiccant coated exchanger for moisture adsorption in a wheel is often quantified using the $\varepsilon_l$ term. For the single-step test, $\varepsilon_l$ is determined by fitting the experimental data to a double exponential model (DEM), according to the associated boundary conditions. The time constants and the mass transfer weighting factors are determined from the curve fitting and the equation relating these factors to the number of transfer units (NTUs). The DEM is used to determine the NTUs along with $\varepsilon_l$ using the associated formula, based on the detailed procedure reported by Fatheieh et al. From the fitted results of the experimental adsorption—desorption data shown in Figure 4a,b, there is close agreement between the experimental and DEM-calculated data, as revealed by the goodness-of-fit results ($R^2 = 0.998$). Other parameters such as the time constant and weighing factors related to the model fitting for the adsorption and desorption processes are listed in Table 2.

### Table 2. Best-Fit DEM Parameters and Weighing Factors Obtained for Step Increase and Decrease for the Inlet Humidity ($Re_{dh} = 26$ and $T_{air} = 23 \, ^{\circ}C$)

| $\Delta RH_{in}$ | $\varepsilon_l$ (ads) | $\varepsilon_l$ (des) | $R^2$ | step type |
|-----------------|---------------------|---------------------|-------|-----------|
| 50 ± 2          | 0.93                | 0.72                | 0.998 | increase  |
| 50 ± 2          | 0.92                | 0.70                | 0.998 | decrease  |

Overall, the normalized humidity profile in Figure 4 (also called the breakthrough curve) indicates a gradual increase in the humidity of the air at the outlet stream until it reaches the humidity in the inlet. Using the DEM, the $\varepsilon_l$ values of the FF-coated exchanger were determined at different angular speeds ($\omega$) ranging from 0.5 to 20. The comparative results against the SPs are shown in Figure 6a,b for both the adsorption and desorption processes, respectively. The results show an increase in $\varepsilon_l$ with an increase in the angular speed, which agrees with previously reported results. Additionally, the value of $\varepsilon_l$ during the adsorption and desorption processes are nearly equal (within uncertainty limits) at identical angular speed, as noted in Figure 6a,b. With reference to the biosiccants, SPs were reported to have comparable $\varepsilon_l$ values at $\omega = 0.5$ for the adsorption ($\varepsilon_l \approx 68\%$) and desorption ($\varepsilon_l \approx 64\%$) processes. Similarly, FF-coated exchangers herein showed comparable $\varepsilon_l$ values for the adsorption ($\varepsilon_l \approx 66\%$) and the desorption ($\varepsilon_l \approx 65\%$) process at similar conditions. The comparable values of $\varepsilon_l$ for the FFs and SPs suggest that FFs have equal potential for use as desiccant coatings for energy wheels. Upon consideration of the relative abundance, mechanical durability, and processing costs of FFs, such fiber-based biomass has even greater potential as a desiccant coating when compared against SPs in coated exchangers.

For the cyclic experimental test, $\varepsilon_l$ was determined for the adsorption and desorption cycles at $\omega = 0.5$, using eqs 3a and 3b as stipulated by the ASHRAE standard.55

$$\varepsilon_l_{\text{ads}} = \frac{\dot{n}(W_t - W_i)}{\dot{m}(W_t - W_i)}$$ (3a)

$$\varepsilon_l_{\text{des}} = \frac{\dot{n}(W_t - W_i)}{\dot{m}(W_t - W_i)}$$ (3b)

In eq 3a, the term $\dot{n}(W_t - W_i)$ is the level of moisture adsorption rate during the adsorption cycle. In eq 3b, the term $\dot{n}(W_t - W_i)$ is the level of moisture desorption rate during the desorption cycle. $\varepsilon_l$ ads and $\varepsilon_l$ des are the $\varepsilon_l$ for the adsorption and desorption processes, respectively.

A comparison of the results for $\varepsilon_l$ obtained after the system reached a quasi-steady-state condition for five different cycles is shown in Figure 7. The results yield a $\varepsilon_l$ value of 72 and 68% for the adsorption and desorption processes, respectively. It is noteworthy that the $\varepsilon_l$ results for the adsorption and desorption processes for the cyclic and single-step tests are in good agreement, within the limits of experimental uncertainty. Also, the $\varepsilon_l$ value is nearly constant for all the cycles, which suggests that the sorption rate is nearly equal for all cycles upon reaching a steady-state condition. A comparison of the $\varepsilon_l$ value of the FF-coated exchanger for both the single and cyclic tests (Figure 8a) reveals that the results obtained for both tests are similar within the limits of uncertainty. Thus, the
DEM model provides a reliable estimate of the latent effectiveness at a defined angular speed.

SPs have been reported to show favorable water sorption properties based on its suitable solvent swelling and water vapor adsorption isotherms. Hence, a comparative analysis of the performance of FF- versus SP-coated exchangers for moisture uptake properties during cyclic tests is shown in Figure 8b. The FFs have a $\varepsilon_l$ value of 72 and 68% for the adsorption and desorption processes, respectively. By comparison, the SP-coated exchanger displays a reduced $\varepsilon_l$ values for the adsorption ($\varepsilon_l = 62\%$) and desorption ($\varepsilon_l = 58\%$) processes, in agreement with the results for the SP-coated system reported by Shakouri et al. The $\varepsilon_l$ values reported herein correspond to a 10% difference in moisture uptake performance between that of FF- and the SP-coated exchanger for the energy wheel test. Because the industrial operation of the energy wheel is based on the cyclic experiment, these results show that FFs have greater moisture uptake overall and better potential as a biodesiccant coating over SPs for energy wheel applications (cf. Figure 8).

**2.3. Structure–Adsorption Properties.** The variable performance of the FF-versus SP-coated exchangers can be accounted for based on the variable biomass components, composition, and structure. The results described above for the complementary material characterization (FT-IR spectroscopy, PSD, SEM, $N_2$ adsorption and water swelling) reveal notable differences in the surface and textural properties of the biodesiccant materials that are mirrored by the adsorption–desorption properties. It is noteworthy that key differences (biopolymer components, composition, and structure) distinguish the variable physicochemical properties of SPs versus FFs. Starch is the major polysaccharide in plants that is composed of two polyglycans (AP and AM), both of which bear surface hydroxyl ($-\text{OH}$) groups for water to bind. These linear and branched polyglycans constitute starch granules of variable sizes and shapes that are organized as semicrystalline and amorphous concentric layers. In contrast, plant-based fibers such as FFs are described as biopolymer-based composites that contain crystalline cellulose microfibris embedded together with various biopolymer components with abundant functional groups: lignins ($-\text{OH}$), hemicellulose ($-\text{OH}$), and pectins ($-\text{OH}, \text{COO}^-$). The performance metrics for the FF- ($\varepsilon_l; 72\%$ adsorption and 68% desorption) and SP-coated ($\varepsilon_l; 62\%$ adsorption and 58% desorption) exchangers are reported in the presence of water vapor using cyclic testing (cf. Figure 8b) under variable RH conditions. The offset in latent effectiveness values between FFs and SPs reflects the variable structure of the biomass-based desiccants and water adsorption properties. The relative magnitude of the $\varepsilon_l$ values for the adsorption (dehumidi-
fication) and desorption (humidification) processes provide a measure of the desiccants’ propensity to “catch and release” water vapor at specified RH and temperature conditions. In general, moisture uptake by desiccants may occur through various modalities; (1) adsorption via surface functional groups; and (2) adsorption within pore domains. The combined effects of (1) and (2) above are demonstrated to play a key role that distinguishes the water uptake properties of the biodesiccants herein. Also, the water sorption capacity can be related to the hydrophile–lipophile balance (HLB) of the FFs and SPs because the uptake properties depend on various factors such as the relative biopolymer content, functional group accessibility, and the textual (SA, PV) properties of the biodesiccant. 64 In the case of the FFs, the surface-accessible functional groups of the fibril composite matrix containing lignins, (hemi)cellulose, and pectins accounts for the HLB character of such materials. Similarly, the SPs are characterized by a supramolecular complex of AM/AP, where the overall HLB character of the biodesiccant relates to its composite structure and overall functional group accessibility.

According to the water swelling results reported in Table 1, the variable hydrophile character and adsorption site accessibility of FFs and SPs can be inferred, along with a comparison of the trends in W, values, where the FFs recorded ca. 5-fold greater W, values relative to the SPs. The foregoing results are corroborated by the FT-IR results, particle size, and textural properties (cf. Figure 4a–d), where support for the presence of (hemi)cellulose, pectins, and lignins is provided, along with differences in the extent of adsorption at the particle grain interfaces for FFs versus SPs. In particular, the greater intensity of the IR band ~1640 cm−1 for FFs reveals its greater uptake of water when compared with SPs, in accordance with the greater hydrophile nature of FFs attributed to its respective biopolymer components. FFs reveal the presence of pectins (~1735 cm−1), lignins, and (hemi)cellulose (~900–1500 cm−1), according to the IR spectral results. Water uptake is inferred via various interactions (H-bonding, van der Waals, and electrostatic) with various active sites (OH, COO−) of the biopolymer fractions. The presence of organophilic (lignins) and hydrophilic (hemicellulose, pectins, and so forth) fractions are known to modify the HLB character of FFs. The latter are mainly amorphous polysaccharides with high O-heteroatom content and relatively high accessibility for water adsorption. 59 The lower composition of lignins is known for FFs 62 and supported herein using complementary methods (cf. Figure 1a), as compared with other hydrophilic biopolymers, such as (hemi)cellulose and pectins. Finally, the difference in the particle size (Figure 1b) and morphology of FFs and SPs (fibrils vs spheroids) is inferred to contribute to water uptake results because the reduced packing efficiency of FFs over SPs contributes to greater adsorption of N2 at the particle grain interface, as observed herein (cf. Figure 1c,d).

The lower degree of water swelling for SPs relative to FFs may indicate the reduced hydrophile character because of possible adhesion between AM/AP 59,50 and/or the presence of trace phospholipidsproteins. 60 In addition to the reduced hydrophile character of AP, adhesion effects among macromolecular components contribute to a lowering of the HLB character and the functional group accessibility, in agreement with the N2 isotherm results (cf. Figure 1c,d) and the parallel attenuation of the water uptake properties. 63 The greater swelling of FFs over SPs in liquid H2O contributes to an apparent increase in the SA because of the surface tension and capillary effects of water, in contrast with the N2 BET SA estimates in Table 1. The FFs are predominantly characterized by surface-mediated (adsorption) processes because of the nonporous nature of the material. The contrasting role of surface- and capillary-driven effects contributes to water uptake for SPs, in agreement with the enhanced textural properties (Table 1). In the case of FFs, the greater uptake of N2 was observed at higher relative pressures (P/P0 ~ 1.0 in Figure 1c,d) that further characterizes the relatively nonporous nature of this material and the key role of adsorption at the particle grain interface sites. Despite the differences in molecular-level properties of N2 and H2O (size and dipolar character) adsorptive probes, the N2 BET results provide estimates of the textural properties of FFs versus SPs. By contrast, results obtained for the water vapor isotherms reveal differences because of its adsorbate size, adhesive interactions, and capillary effects, as evidenced by the contrasting results reported for the SPs. 64 The textural properties of the biodesiccants in the present study are further supported by the single-step change and cyclic test results, according to differences in the rate of adsorption/desorption for FFs versus SPs. Faster desorption rates for the FF-coated exchanger (cf. Figure 4d) suggest that water uptake in this material is largely mediated via surface processes at the particle grain interface, as noted above. This is consistent with the surface tension properties of H2O because the surface-bound H2O is desorbed more readily relative to pore-bound H2O. 61 Constant W in the breakthrough curves (cf. Figure 4c) at the initial stages (t ~ 7 S) provides support that SPs have variable textural features over FFs, as evidenced by greater moisture adsorption at such conditions. While the composite structure of FFs is characterized by limited textural porosity, it has a complex fibril morphology with greater particle size and abundant functional groups for water adsorption at the particle surface that accounts for its unique properties over SPs, as evidenced by the materials characterization results for this study.

3. CONCLUSIONS

The adsorption–desorption performance of FFs was evaluated for a potential biodesiccant coating for an air-to-air exchanger in a laboratory-scale test facility. Raw FFs were ball-milled and mechanically ground, along with complementary spectroscopic (FT-IR and SEM) and adsorption-based characterization to elucidate the biodesiccant structure–function properties. The performance of the FF-coated exchanger for moisture uptake was revealed using single-step and cyclic test methods, where the latent effectiveness (e) was compared against that of a SP-coated exchanger. This comparison provided an improved understanding of the biodesiccant structure–adsorption property relationship for FFs and SPs. The overall results reveal that the single-step fitting method accounts for the experimental latent effectiveness from the cyclic tests within the experimental uncertainty limits at controlled conditions. A comparison of the SP- and FF-coated exchangers reveal notably greater e-values for FFs (72 and 68%) versus SPs (62 and 58%) for the adsorption and desorption processes, respectively. The disparity in the performance of the two biodesiccants was explained in terms of the differences in the relative role of surface- and capillary-driven adsorption because of the key role of particle grain adsorption sites for FFs, in agreement with the limited textural properties of these materials. The swelling results in liquid water reveal greater adsorption site accessibility for the FFs, as evidenced by the
fivefold greater swelling over the SPs. The combined effect of adsorption site accessibility and the hydrophilic character of the FFs relates to the biopolymer composition and structure, in agreement with the spectroscopic and isotherm results. The biodesiccant characterization by SEM, PSD, and N₂ adsorption reveals that FFs possess limited textural porosity (Table 1), as compared with SPs. Water uptake is largely surface-mediated for FFs, whereas adsorption of water by SPs is largely driven by surface- and capillary-driven effects. The cyclic test results for the FF-coated exchanger over the multiple cycles showed that the adsorption−desorption process occurred at equilibrium conditions because latent effectiveness was equal across multiple cycles. Based on the results reported herein, FFs represent a promising and sustainable alternative biodesiccant coating for air-to-air heat exchangers in energy wheel applications, where the latent effectiveness rivals the properties of SPs biomass and other conventional solid desiccant systems (e.g., SGs).

From a techno-economic perspective, the relative biodesiccant performance of FFs and SPs reveal that FFs are a promising alternative material in view of their abundant and sustainable supply from waste agricultural biomass.

4. EXPERIMENTAL SECTION

4.1. Materials, Chemicals, and Treatment. The FFs obtained from Biolin Research Inc. (Saskatoon, SK, Canada) were subjected to grinding, ball milling, and sieving prior to characterization, and moisture uptake studies were carried out in the test facility described previously. The FF is made up of ∼70% cellulose, 15% hemicellulose, 2−5% pectin, and 5−8% lignin. The extracted raw FFs were ground by mixing with zirconium oxide (ZrO₂) ceramic balls (6−10 mm in diameter) in a stainless steel milling jar. The rotation speed of the disk and milling jar was set to 450 rpm, where the jar was alternately rotated in the forward and reverse directions at intervals of 2 min. The ground FF sample was sieved through a mesh size #120 sieve to achieve uniform particle sizes of ∼125 μm. Herein, the physicochemical properties of FFs were compared against results obtained for SPs in a previous study.

The spectroscopic-grade potassium bromide (KBr) and wire mesh sieve [USA standard Testing Sieve; no. 120 (125 μm)] were purchased from Sigma-Aldrich (ON, Canada).

4.2. Materials Characterization. The physicochemical properties of the flax (FFs) material were characterized using similar techniques that align with the previous report on SPs, as outlined below.

4.2.1. FT-IR Spectroscopy. FT-IR spectra of the FFs sample were recorded using a Bio-Rad FTS-40 spectrophotometer in the diffuse reflectance mode using KBr as the diluent, where the biomass sample constituted ∼80% (w/w) of the total sample. Multiple scans (n = 64) were taken for each sample from 4000 to 400 cm⁻¹ spectral range at a resolution of 4 cm⁻¹.

4.2.2. Particle Size Analyzer. The PSD of the sample was measured on a Mastersizer S particle size analyzer (Malvern), where the samples were analyzed as dry powders.

4.2.3. N₂ Gas Adsorption Isotherms. The N₂ gas adsorption test of the FFs powder was performed on a Micrometrics ASAP 2020 S2 instrument (Norcross, GA) to estimate the BET accessible SA, pore width (PW), and PV distribution of the materials.

4.2.4. Scanning Electron Microscopy. SEM images were obtained using FEG-SEM SU6600 instruments at an...
Table 4. Operating Conditions Used for Both Transient and Cyclic Tests Experiments

| $Q_s$ (L/min) | $V_i$ (m/s) | $Re_{fl}$ | $T_{air}$(°C) | $RH_{fl}$ (%) | $RH_{humid}$ (%) | $\Delta RH$ step (%) |
|--------------|-------------|-----------|---------------|--------------|-----------------|------------------|
| 15 ± 1       | 0.050 ± 0.001 | 26 ± 2  | 23 ± 0.5  | 7 ± 2       | 50 ± 2          | 43 ± 2           |

accelerating voltage of 15 kV. The coated FF membrane was mounted on aluminum stubs with a conductive carbon tape, where a platinum surface coating was achieved using an ion sputter coater prior to imaging.

4.2.5. Gravimetric Swelling Tests. Gravimetric swelling of the desiccant (FFs) was performed in water as the solvent, where the solid samples (~20 mg) were equilibrated in Millipore water (7 mL) for 48 h. Solvent swelling ($S_{W}$) was determined using eq 4, where $W_f$ and $W_d$ are the weights of the wet and dry samples, respectively.

$$S_W = \frac{W_f - W_d}{W_f} \times 100\%$$

(4)

5. TEST FACILITY FOR TRANSIENT TESTING

5.1. Preparation of a Parallel Plate Exchanger. Rectangular aluminum plates (Al-3003 sheets) with the dimensions (20 cm × 9 cm × 0.65 mm) were obtained from McMaster-Carr Supply Company (USA). The ball-milled FFs were coated onto the surface of the Al sheets using a unique sieving method developed in-house. The FFs were screened to ensure particle sizes were relatively uniform ($\leq 125 \mu m$) using a 120 mesh US standard sieve. The required amount of FF (measured) was then applied on the surface of a thin (0.035 mm) adhesive-aluminum tape attached to aluminum plates using deposition via controlled sieving. After the coating process, the flax-coated Al plates were left to dry in open air for 24 h, where the coating was observed to retain its original uniformity without any visible bulging or perforation. Subsequently, a small-scale parallel plate exchanger (20 cm × 10 cm × 7 cm) was assembled using 16 Al sheets coated with flax, as shown in Figure 3. Parameters regarding the average mass (g) and mass per unit plate area (g/cm²) of the FF coated on the small-scale exchanger were comparable to the values for similar plates that were coated with SPs with desiccant-to-matrix mass ratios reported as 0.64 and 0.69, respectively. Additional parameters for the FF-coated small exchanger are highlighted in Table 3.

5.2. Test Apparatus and Experimental Procedure. A schematic diagram of the test facility is shown in Figure 9, where it consists of two units: the air stream preconditioning unit and the test unit. The preconditioning unit is composed of a supply air system, where a compressor is in line with a dehumidifier. The test unit consists of a test section where the small-scale exchanger is located, humidity sensors and thermocouples at the inlet and outlet of the test section, a linear actuator unit which slides the exchanger between humid and dry air streams. In this facility, the exchanger can be exposed to single and periodic step changes in humidity. The maximum uncertainty in temperature and relative humidity measurements are ±0.2 °C and 1%, respectively. The principle and procedures of the experiments are detailed in Section 5.3. Further explanation of the facility and the roles of each component can be found in previous reports.

5.3. Single-Step and Cyclic Test Procedure and Operating Conditions. The test facility (Figure 9) was used to test the performance of the FF-coated desiccant onto a small-scale exchanger using a single-step change. Table 4 shows the test conditions and flow rates used for the testing. The general procedure involves three steps: (1) preconditioning; (2) humidity step change; and (3) transient measurements. During the preconditioning of the regeneration step, air streams at variable temperature and humidity (RH = 50%) were passed through the ducts for a minimum of 1 h before the step change in order to achieve steady-state conditions. For dehumidification testing, dry air (RH < 9%) was used for preconditioning of the exchanger until steady-state conditions were reached. Herein, steady-state conditions are defined by the temperature and humidity variations within the sensor uncertainty for a period of 1 h. For the humidity step change, the inlet humidity was changed (<1 s) by automatically sliding the exchanger rapidly from the dry to humid air streams.

For the regeneration tests, after preconditioning the exchanger with humid airflow, the exchanger was switched to the dry air stream. As a result, the dry airflow passes through the exchanger where the desiccant starts to desorb the adsorbed water vapor. In the last step (transient measurements), the temperature and humidity at the inlet and outlet streams were recorded until the outlet humidity reached the inlet humidity. The acquired data set represents the transient response of the exchanger.

In the cyclic experiment, the exchanger was exposed to series of adsorption/desorption cycles until the outlet air streams (air ducts 3 and 4) reached a steady-state condition. A dynamic steady state is reached using the exchanger when the difference between the average latent effectiveness of two cycles is below 1%, a relative difference that is below the experimental uncertainty limit. During one experimental cycle, 120 s was used as the period which is equal to 0.5 rpm, and other conditions related to the experimental step are shown in Table 4.

AUTHOR INFORMATION

Corresponding Author

Lee D. Wilson – Department of Chemistry, University of Saskatchewan, Saskatoon, Saskatchewan S7N 5C9, Canada; oicid:0000-0002-0688-3102; Phone: +1-306-966-2961; Email: lee.wilson@usask.ca; Fax: +1-306-966-4730

Authors

Wahab O. Alabi – Department of Mechanical Engineering, University of Saskatchewan, Saskatoon, Saskatchewan S7N 5A9, Canada
Abdalla H. Karoyo – Department of Chemistry, University of Saskatchewan, Saskatoon, Saskatchewan S7N 5C9, Canada; oicid:0000-0002-6410-0241
Easwaran N. Krishnan – Department of Mechanical Engineering, University of Saskatchewan, Saskatoon, Saskatchewan S7N 5A9, Canada
Leila Dehabadi – Department of Chemistry, University of Saskatchewan, Saskatoon, Saskatchewan S7N 5C9, Canada
Carey J. Simonson – Department of Mechanical Engineering, University of Saskatchewan, Saskatoon, Saskatchewan S7N 5A9, Canada; oicid:0000-0003-3331-9184

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.0c00762
Notes
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