Water Vapor Adsorption—Desorption Behavior of Surfactant-Coated Starch Particles for Commercial Energy Wheels

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ABSTRACT: This study reports on the adsorption (dehumidification)—desorption (humidification) behavior of cetylpyridinium bromide (CPB) coated starch particles (SPs), denoted as SP-CPB, as a potential desiccant material for air-to-air energy exchangers. CPB is a cationic surfactant with antibacterial activity that can be used to modify the surface properties of SPs, especially at variable CPB loading levels (SP-CPB0.5, SP-CPB2.5, and SP-CPB5.0, where the numeric suffix represents the synthetic loading level of CPB in mM). The SP-CPB0.5 sample displayed optimal surface area and pore structure properties that was selected for water sorption isotherm studies at 25 °C. The CPB-coated SPs sample (SP-CPB0.5) showed an improved water vapor uptake capacity compared to unmodified starch (SPs) and other desiccant systems such as high amylose starch (HAS15) and silica gel (SG13). Single-step and cyclic water vapor sorption tests were conducted using a small-scale exchanger coated with SP-CPB0.5. The calculated latent effectiveness values obtained from direct measurements using cyclic tests (65.4 ± 2%) agree closely with the estimated latent effectiveness from single-step tests (64.6 ± 2%) at controlled operating conditions. Compared to HAS15- and SG13-coated exchangers, the SP-CPB0.5-coated exchanger performed much better at controlled operating conditions, along with improved longevity due to the CPB surface coating. The presence of CPB did not attenuate the uptake properties of native SPs. Latent effectiveness of SP-CPB0.5-coated exchanger was enhanced (5–30% higher) over that of the SG13 or HAS15-coated exchangers, according to the wheel angular speed. This study reports on a novel and sustainable SP-CPB0.5 material as a promising desiccant coating with tunable uptake and surface properties with potential utility in air-to-air energy exchangers for ventilation systems.

INTRODUCTION

Indoor air quality (IAQ) plays a key role on the mental and physical health of occupants, affecting their comfort and productivity.1 The IAQ and comfort in buildings mainly depend on the air temperature, humidity, and outdoor ventilation rate within indoor air environments. Different space heating/cooling methods are available to maintain the indoor air temperature within the optimal comfort zone.2 Considering human health and comfort, the relative humidity (RH) levels between 30 and 60% are recommended.1 Researchers are currently focused on developing new technologies to reduce the energy footprint of heating, ventilation, and air conditioning (HVAC) systems, while maintaining the required level of IAQ and comfort.

There is continued interest in the study of desiccant materials due to their critical role as functional coatings in HVAC systems, especially for applications in air-to-air energy exchangers (AAEEs). The central importance of desiccants and their water vapor adsorption—desorption properties in AAEEs relates to the improvement in energy efficiency of HVAC systems. The global energy footprint and greenhouse gas emissions related to heating and cooling of residential and commercial buildings is substantial. The heating and cooling of buildings (commercial, residential, and industrial) accounts for 30–50% of energy consumption and greenhouse gas emissions in Canada3 and the EU.5 Furthermore, the demand for cooling is expected to increase almost exponentially (by 30 times over this century) due to climate change and increased demand in developing countries.6 Currently, about 60% of energy consumed by residential buildings, while 54% of energy consumed by commercial buildings are used for space heating, cooling, and ventilation in Canada.7 Thus, HVAC systems account for 15–30% of global energy consumption. It follows that activities related to the research and development of improved desiccant coatings can have significant impact on the efficiency of AAEEs and thereby lower the energy consumption and greenhouse gas (GHG) emissions.
Among various types of AAEEs, energy wheels are widely used in large-scale commercial applications because of their unique capability for the transfer of both heat and moisture between air streams. Energy wheels have two main components: the substrate and desiccant. The substrate is typically a thin aluminum sheet that is crimped and wound to form a perforated wheel to allow airflow and enable sensible heat transfer through the wheel architecture. The desiccant is a hygroscopic material that is coated onto the substrate to allow moisture transfer (adsorption/desorption of water vapor) due to the gradient of water vapor pressure between the airflow streams and the surface of the desiccant. Therefore, research on the nature of the desiccant and strategies to enhance the desiccant properties is a key area of research and development.

Desiccants can be selected from inorganic materials such as silica gels (SGs),10–13 zeolites (molecular sieves),13,14 activated alumina,15–17 to organic desiccants such as porous/activated carbons.12,17,18 Also, hybrid substrates such as synthesized silica gel-composite materials19–21 and metal–organic frameworks (MOFs) have been reported.22–26 Research on alternative desiccants such as starch, wheat, cassava, corn grit, and durian peel suggest that agriculture-based biomass possess unique water adsorption–desorption capabilities with additional benefits of low cost, high abundance, and sustainable nature.27–29 Recently, Fathieh et al.30,31 conducted a sorption study on high amylose starch (HAS15) using the component testing of an energy wheel, where the performance of HAS15 and mesoporous silica gels (SG13) with variable particle sizes were compared (commercial solid desiccants). Latent effectiveness is the one of the most important parameters to quantify the moisture transfer performance of air-to-air energy exchangers.32 It is defined as the ratio of the actual moisture transfer rate to the thermodynamic maximum moisture transfer rate and therefore is restricted to have values between 0 and 1.35,33 It was reported that the latent effectiveness of the HAS15-coated exchanger was 2–13% greater than a commercial desiccant (SG13-coating) for similar loading levels and at variable operating conditions. In a follow-up study on a HAS15-coated small-scale exchanger, Dehabadi et al.34 reported on the effects of air flow on the water vapor adsorption–desorption processes using a single-step transient testing at variable air flow rates and temperatures. The results indicated an acceptable exchanger performance for the HAS15-coated exchanger over a range of temperatures (22.5–37.5 °C) and flow rates (10–50 L/min) that further reveal the potential of high amylose starch-based desiccants. More recently, Hossain et al.35 compared the performance of a coated exchanger that used starch particles with high amylopectin content (SP15) that was compared against HAS15- and SG13-coated exchangers with comparable desiccant loadings. Among these desiccants, the weight-based moisture recovery (g of water vapor/g of desiccant) of SP15 (0.152) was 3-fold over HAS15 and 6-fold greater over SG13 (0.026). The impressive moisture recovery, along with ~98% regeneration of the SP-based desiccant during the vapor desorption process relates to the unique biopolymer structure such as its hydrophilic character, structural flexibility, and accessibility of the biopolymer surface –OH groups. The favorable balance between the adsorption and desorption properties reported for the SPs was referred to as the “Goldilocks effect,” as evidenced by comparable differences in the enthalpy for the adsorption/desorption (∆H_ads/∆H_des) process with water vapor.36 The magnitude of ∆H_ads/∆H_des below 150 kJ/mol was indicative of a physisorption process,36 indicating that the SP-based desiccant was highly suitable as a coating material in AAEE systems.

While the adsorption–desorption properties were deemed favorable under dynamic flow conditions for SPs, the potential longevity of starch under moderate temperature and humidity conditions36,37 was a potential concern for AAEEs, especially

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Figure 1. TEM images of native starch particles (SPs) and cetylpyridinium bromide-coated SPs (SP-CPB0.5). (a) 80k× magnification and (b) 150k× magnification of SPs. (c) 100k× magnification and (d) 150k× magnification of SP-CPB0.5. The layer-by-layer amorphous and crystalline domains are indicated with dotted ovals.
for multiple adsorption-desorption cycles over extended time intervals. To address this concern and characterize the physicochemical properties of surface-modified SPs, a study\(^\text{38}\) was undertaken to explore the role of surface modification of SPs using various dopant levels (0.5, 2.5, and 5.0 mM) of cetylpyridinium bromide (CPB) relative to unmodified SPs. The respective dopant levels of CPB surfactant onto the surface of SPs correspond to the monolayer surface coverage (SP-CPB0.5), while elevated CPB levels in the case of SP-CPB2.5 and SP-CPB5.0 concur with a multilayer surface coverage reported elsewhere.\(^\text{38,39}\) The water sorption properties of SPs in liquid water at variable CPB dopant levels did not significantly attenuate the water sorption properties, as compared with unmodified SPs. By contrast, the longevity of SPs was improved for SP-CPB0.5, as evidenced by the enhanced antimicrobial activity of CPB toward a range of bacterial strains, as compared with unmodified SPs.\(^\text{38}\) The aim of this paper is to further our research efforts toward new biopolymer desiccants\(^\text{40-42}\) with improved sorption capacity, adsorption/desorption regeneration, and temporal stability by testing surfactant-coated SPs. Herein, SP-CPB0.5 (surfactant concentration = 0.5 mM) was selected based on a previous optimization study. The current study reports on the comparison of the latent performance of SP-CPB0.5 as a desiccant with those of two types of desiccants (SG13 and HAS15) using sorption/desorption tests of water vapor with a small-scale energy exchanger developed in-house.\(^\text{43}\)

Several objectives are pursued in the current study: (1) to compare the water vapor adsorption—desorption properties of surfactant-modified SPs relative to native SPs desiccant coatings and (2) to understand the structure—function relationship of the CPB-coated SPs at typical conditions relevant to AAEEs. In this study, we highlight the development of a sustainable and tunable surface-modified starch-based desiccant with unique vapor adsorption (dehumidification) and desorption (regeneration) properties. Moreover, it will be shown that the use of facile surfactant doping via deposition of CPB onto SPs does not attenuate the unique vapor adsorption—desorption properties according to the remarkable “Goldilocks effect” previously reported for unmodified SPs.

The latter is considered as a hallmark feature for key technological advancements in the design of sustainable desiccant materials to enable efficient energy harvesting in energy wheel applications.

### RESULTS AND DISCUSSION

As noted above, Karoyo et al.\(^\text{38}\) carried out a systematic study of the surface modification of starch particles (SPs) with CPB in an effort to determine the optimal level of monolayer surfactant doping (cf. Figure 9 in ref 35 or Figure 1 in ref 38). According to the trends in solvent swelling results in water, CPB-doped SPs revealed optimal swelling for surface-modified SPs at a loading concentration of 0.5 mM for the CPB surfactant (SP-CPB0.5). The optimal surfactant loading was further aided by other complementary thermoanalytical and spectroscopic methods, as described elsewhere.\(^\text{38,39}\) In the present study, transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), and water vapor adsorption isotherms were used to gain further insight into the “Goldilocks effect”\(^\text{35}\) through the study of the dynamic vapor adsorption properties of SPs modified with CPB using a small-scale coated exchanger developed in-house.\(^\text{41}\)

**TEM.** The TEM images of the SPs and SP-CPB0.5 are shown in Figure 1a,b, respectively. Figure 1a reveals the semicrystalline nature of the SPs which contains alternating layer-by-layer (marked with dotted ovals in the image) amorphous and crystalline domains arranged in concentric rings.\(^\text{43}\) Furthermore, the shape of the SPs are composed mostly of spherical granules, with a few distinctly elongated granules.\(^\text{34}\) In contrast to the morphology of SPs (Figure 1a), the TEM image of the SP-CPB0.5 (Figure 1b) is characterized by larger and darker granules without the apparent layer-by-layer structural features. The apparent difference in morphology of the SP-CPB0.5 relative to that of unmodified SPs is related to the presence of a bound CPB at the surface of SPs. The surface-bound CPB surfactant may contribute to alteration of the hydrophilic—lipophilic balance (HLB) of the interface, in agreement with the larger granule size and agglomeration of the CPB-doped SPs due to hydrophobic interactions, as noted in Figure 1b.\(^\text{35,46}\)
XPS. X-ray photoelectron spectroscopy (XPS) is a useful method for gaining information regarding the state of chemical structure of biopolymer materials. In this study, XPS was used to investigate the effect of the CPB surfactant on the structure of the SPs. Figure 2 shows normalized XPS spectra of CPB, SP-CPB0.5, and SPs. The peaks at \( \sim 282.5 \) and \( \sim 284 \) eV reveal the presence of \( \text{sp}^2 \) and \( \text{sp}^3 \) orbitals of C 1s, respectively. These orbitals are related to the C–H and C–C and C–O bonds of the SPs. Also, the presence of O 1s at \( \sim 530 \) eV and N 1s at \( \sim 398 \) eV relate to the heteroatoms of starch and the peptide components in the starch granule, respectively. Further survey of the XPS spectra in Figure 2 reveals the presence of Br 3d\(_{5/2}\), Br 3d\(_{3/2}\), and Br 3p signatures at \( \sim 66 \), \( \sim 68 \), and \( \sim 184 \) eV, respectively. These peaks account for the presence of the Br–C bonds in the CPB sample that provide further support of the adhesive binding interaction of the CPB surfactant with the SPs, in line with the TEM results in Figure 1b. In particular, the signature at \( \sim 66 \) eV corresponds to the Br 3d that provides evidence of the CPB coating on the starch. It should be noted that the low intensity of this band relates to the relatively small amount of CPB (0.5 mM) used in the coating.

Water Vapor Adsorption. Figure 3 shows the water vapor adsorption isotherms of the native SPs (a) and SP-CPB0.5 (b) samples at 25 °C, where the results of the sample of SPs coated at higher CPB loading (SP-CPB5.0, Figure 3c,d) are compared. In the stacked plots in Figure 3d, the water vapor uptake capacity (w/w %) for the SP-CPB5.0 sample (36.5%) is comparable to the value for the SP-CPB0.5 sample (36.0%), whereas lower values are reported for native SP samples (33.5%). The relative similarity of the SP-CPX (X = 0.5 and 5) above may be related to the comparable surface accessibility of the active hydrophilic sites in spite of variable CPB loading. In general, the isotherm results in Figure 3 reveal type II isotherm profiles that are characterized as macroporous materials with unrestricted monolayer–multilayer adsorption profiles. At low relative pressures (\( P/P_0 \)), near 0.2 (~10 wt %), the water molecules can be adsorbed at the surface of the native and coated SPs samples that results in the formation of a monolayer adsorption profile. At \( P/P_0 > 0.8 (>20 \text{ wt %}) \), the formation of bilayer and/or multilayer surface-bound water profiles are anticipated and the total water vapor capacity is reached at \( P/P_0 = 0.95 \). The greater uptake capacity at high \( P/P_0 \) values indicate a strong relation between adsorbent porosity and vapor uptake properties. The uptake capacity values reported herein meet values reported by Furukawa et al. where the wt % values ranging between ~8 and 68 were reported for zirconium-based MOFs and related materials. It is worth noting that the adsorptive interactions of water with solid bioadsorbent materials is governed by several factors: (i) accessible surface functional groups, (ii) the polarity and HLB of the bioadsorbent, and (iii) the presence of amorphous and crystalline domains of the adsorbent that relates to the relative amylose (linear) versus amylopectin (branched) content of starch.

In the case of native SPs, it is noteworthy that water vapor can access the active hydrophilic starch domains. By contrast, the surface –OH functional groups are less accessible for the CPB-coated SPs. In the case of CPB-coated SPs, the outer surface is characterized by steric effects due to the bound surfactant on the starch surface and contributions from particle aggregation, as described in the TEM and XPS results. The trends in the water vapor uptake capacity herein (i.e., SP-CPB5.0 (36.5%) \( \geq \) SP-CPB0.5 (36.0%) > SPs (33.5%)) reveal that the interaction of water vapor with the starch samples is achieved via both adsorption and absorption (sorption)
processes. The foregoing indicates that the coated SP samples (i.e., SP-CPB0.5 and SP-CPB5.0) contain variable hydrophilic sites (ionic CPB domains or dipolar starch sites) that slightly favor the uptake of water vapor over the native SPs. The effect of the surface-bound CPB is to afford additional pores or hydrophilic sites that enhance the vapor permeability of the CPB-coated starch, in line with the results for cassava starch hydrophile sites that enhance the vapor permeability of the SP-CPB systems. The high water uptake capacities and effective moisture adsorption and desorption properties as described above and elsewhere.\(^{54}\)

It is important to note that the mechanism of surface binding for liquid water versus gaseous water onto the surface of starch may differ markedly since the latter is largely driven by the diffusion of water molecules and their subsequent absorption within the pores and surface sites. Thus, the self-assembly of CPB in the SP-CPB0.5 and SP-CPB5.0 samples provide favorable conditions for the adsorption of water vapor, where similar vapor uptake capacities are observed for these systems. The high water uptake capacities and efficient water vapor adsorption/desorption rates are further detailed herein (vide infra). The water vapor adsorption processes for energy harvesting applications should be characterized by favorable enthalpies of adsorption (\(\leq 50 \text{ kJ} / \text{mol}\)) to afford easy regeneration of the desiccant during the desorption process, in accordance with the “Goldilocks effect” described elsewhere.\(^{53,54}\)

It is concluded from the above results that the adsorption of CPB occurs at the surface sites of the SPs that may affect the resulting particle size distribution (due to agglomeration), \(\zeta\)-potential values, and HLB of the starch particles. These physiochemical properties are known to influence the water vapor adsorption properties\(^ {39}\) and are supported by the adsorption isotherms, XPS, and TEM results herein. Among the variable surfactant loadings onto the SPs, the SP-CPB0.5 system provides optimal conditions for effective moisture adsorption and desorption properties as described above and elsewhere.\(^ {39}\)

**Single-Step Experiment Results.** The normalized humidity ratio, or the transient response of the small-scale coated exchanger, is defined as the ratio of the difference in humidity ratio of the exchanger at any time to the maximum difference during the test. It can be calculated using eq 1, where \(W\) is the humidity ratio (\(\text{kg}_w / \text{kg}_w\)) of air stream at the exchanger outlets.

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

The humidity ratio of air streams at the exchanger inlet and outlet was calculated from the temperature and relative humidity measurements.\(^ {55}\) The transient dehumidification and regeneration responses (break-through curve) of the SP-CPB0.5-coated small-scale exchanger, \(W\), versus time are shown in Figure 4. During the dehumidification (adsorption), \(W\) slowly increases from 0 at time 0 s to 0.95 at about 300 s (Figure 4a). This indicates that the SP-CPB0.5-coated exchanger reached about 95\% of its equilibrium moisture content during the initial 300 s. As the test continued, the humidity level of supply and exhaust air streams can reach the same value of moisture content (\(W = 1.0\)). The reverse trend occurs during the desorption process, where \(W\) slowly decreases from 1.0 at the beginning of the step change to about 0.05 after 600 s.

The dehumidification and regeneration latent effectiveness (\(\epsilon\)) of an energy wheel can be estimated from an analysis of their normalized humidity response trends.\(^ {56}\) First, the number of mass transfer units (NTU\(_m\)) is predicted by fitting a double exponential model to the normalized humidity response curves. Then, the latent effectiveness of counter-flow energy exchanger for the balanced supply and exhaust air streams can be estimated using eq 2. Detailed expressions and methods to determine the latent effectiveness from the transient humidity response were reported in a previous work.\(^ {56}\)

\[
\epsilon = \frac{\text{NTU}_m}{1 + \text{NTU}_m}
\]

Figure 5 shows the estimated latent effectiveness values from the single-step change test results at various wheel angular speeds (\(\omega\)). The results indicate that the latent effectiveness of wheel increases with increase in angular speed, which is expected from the literature.\(^ {57,58}\) The moisture transfer rate between the desiccants and air streams is maximum at the beginning of each adsorption/desorption cycle, thus a shorter cycle period (higher angular speed) will result in a higher effectiveness. At low angular speeds, the desiccant is exposed to the air stream for long durations and gradually the desiccant reaches its capacity; hence, the moisture transfer rate decreases, which reduces the latent effectiveness. The estimated latent effectiveness during adsorption (dehumidification) is slightly higher than during desorption (regeneration) at all of the angular speeds investigated. The difference in the latent effectiveness decreases as the angular speed (\(\omega\))

![Figure 4. Normalized humidity response (break-through curve) of SP-CPB0.5 small-scale exchanger during (a) dehumidification (adsorption) and (b) regeneration (desorption) (\(\Delta RH = 40\%\), maximum uncertainty in the experimental data is \(U_W = \pm 0.05\)).](image)
Cyclic Experiment Results. The latent effectiveness of energy wheels can also be determined by conducting cyclic experiments. The operating conditions of an energy wheel using the cyclic test facility are identical to the single-step tests reported in Table 2. To compare with previous reported work, the cyclic experiments were conducted at an angular wheel speed of 0.5 rpm. The relative humidity at the exchanger outlet is measured continuously during the adsorption and desorption cycles. The latent effectiveness is determined after exchanger has reached a periodic steady state, using eqs 3 and 4

\[ e_{L-ad} = \frac{m(W_1 - W_2)}{\bar{m}W_1} \]

\[ e_{L-reg} = \frac{m(W_4 - W_2)}{\bar{m}W_1} \]

where \( \bar{m} \) is the mass flow rate of dry air (kg/s) and subscripts 1–4 refer to sensor in ducts 1–4 in Figure 10. In eq 3, \( m(W_1 - W_2) \) is the moisture adsorption rate for the exchanger during the adsorption or dehumidification cycle; in eq 4, \( (W_4 - W_2) \) is the moisture desorption rate from the exchanger during the desorption or regeneration cycle.

Figure 6 shows the absolute water content profile of the SP-CPB0.5-coated exchanger for four consecutive cycles after 1 h upon reaching a periodic steady-state/equilibrium condition. The results show that during the first half of each cycle, when humid air stream passes through the exchanger, the desiccants absorb moisture, and the air becomes dry at the exchanger exit (dehumidification/adsorption cycle). In the second phase of each cycle, the desiccants start releasing adsorbed water vapor when dry air passes through the exchanger, resulting in an exit air stream that becomes more humid (regeneration/desorption cycle). This process continues during every adsorption–desorption cycle. The results also reveal that the CP-CPB0.5-coated exchanger can transfer similar level of water vapor during dehumidification (adsorption) and regeneration (desorption) cycles. This may indicate that the surfactant-coated desiccants maintain their physiochemical properties during the cyclic experiments. However, the durability of the material in multiple cycles over extended time warrant further investigation in future studies.

The average dehumidification and regeneration effectiveness of individual cycles after reaching a steady state can be calculated from the average humidity ratio (water content) of the outlet and inlet air streams. Detailed expressions and methods to calculate the average latent effectiveness for cyclic testing are reported elsewhere.6 It is important to mention that the decrease/increase of RH values during the first 8 s of each dehumidification/regeneration cycle arises from the time delay in the response of the RH sensors.60,61 Therefore, these values were not accounted for in the effectiveness calculation of the cyclic tests. The maximum and minimum RH values during dehumidification and regeneration cycles were taken as the initial reference points, respectively. The latent effectiveness of the exchanger for each adsorption and desorption cycle from 101 to 106 are calculated and shown in Figure 7.

Table 1. Masses and Coating Techniques of the Various Desiccants Coated on Small-Scale Exchangers

| desiccant      | mass of coating (g) | desiccant (mg/cm²) | desiccant/matrix mass ratio | technique | reference |
|----------------|---------------------|--------------------|-----------------------------|-----------|----------|
| SP-CPB (0.5)   | 3.34 ± 0.02         | 0.696 ± 0.005      | 0.54                        | Sieving   | this work |
| SP15           | 3.43 ± 0.02         | 0.714 ± 0.005      | 0.69                        | Sieving   | 9        |
| HAS15          | 3.20 ± 0.02         | 0.667 ± 0.004      | 0.68                        | Spraying  | 6        |
| SG15           | 3.12 ± 0.02         | 0.650 ± 0.004      | 0.68                        | Spraying  | 6        |
Results are in agreement with the estimated effectiveness from the single-step experiments, where the latent effectiveness of the regeneration was ca. 2% higher than that of the dehumidification step. Figure 8 compares the latent effectiveness of SP-CPB0.5 for a coated small-scale exchanger calculated from the cyclic test (0.5 rpm) that was estimated from the single-step test (breakthrough curves). The average latent effectiveness from cyclic tests is 65.4 ± 2 and 70.7 ± 2% for the dehumidification and regeneration cycles, respectively. The estimated values from single-step tests, which were slightly less than those from the cyclic tests, are 64.6 ± 2 and 68.2 ± 2% for the dehumidification and regeneration step changes, respectively. Results clearly indicate that the calculated values from the cyclic testing agree with the estimated values from the single-step testing within the uncertainty of the measurements. It means that both cyclic and single-step tests can accurately estimate the steady-state performance of the energy wheels in a laboratory-scale facility. On the other hand, the cyclic test is more practical since it is a direct measurement of the effectiveness since there is no need to predict the latent effectiveness of the wheel by using data fitting and correlations.

**Latent Effectiveness Comparison.** Figure 9 compares the estimated values of the latent effectiveness for various desiccant-coated wheels at the operating condition given in Table 2 and different wheel angular speeds (ω), where ω ∼ 0.5 rpm and dehumidification: SP-CPB0.5 (68 ± 2%) > SP15 (64 ± 2%) > HAS15 (46 ± 2%) > SG13 (38 ± 2%). Results show that SP-coated wheels display the best performance for the entire tested angular speed (ω) range. The surfactant-coated starch desiccant performs best during humidification (adsorption). By comparison, the surfactant-coated starch desiccant (SP-CPB0.5) performs best during the dehumidification (adsorption), whereas the native starch desiccant (SP15) performs best during regeneration (desorption).

It is well-known that in addition to the sorption uptake capacity of the desiccants, the kinetic rate of sorption is a parameter of key importance in the performance of energy wheels. Although SP-CPB0.5 had a greater water uptake capacity over SP (Figure 3), it seems that the presence of a surfactant coating may result in steric effects on the starch particle surface with a decreased desorption rate. Thus, the regeneration of SP-CPB0.5 becomes slightly attenuated relative to SP15, resulting in a lower latent effectiveness of the SP-CPB0.5-coated exchanger as compared with the SP15-coated exchanger.

The results also show that the estimated latent effectiveness of the SP-CPB0.5-coated wheels, especially at low angular speed (ω < 1), was slightly lower (ca. 2−4%) during the regeneration cycles than during the humidification cycles. This can be related to capillary condensation within the pores of desiccants, which often requires more energy input during regeneration of desiccants than the dehumidification process. The estimated latent effectiveness increases with increasing angular speeds. Thus, it can be concluded that the system may have greater efficiency at typical operating wheel angular speeds (ω ∼ 10−20 rpm) relative to lower values for ω reported above.

It is important to note that the break-through curves cannot be expressed according to a per unit mass of desiccant since the estimated latent effectiveness is mass dependent. As shown in Figure 9, the average steady-state latent effectiveness of adsorption (dehumidification) and desorption (regeneration) cycles.

![Figure 7. Average steady-state latent effectiveness of adsorption (dehumidification) and desorption (regeneration) cycles.](image1)

![Figure 8. Comparison of latent effectiveness from single-step and cyclic experiments.](image2)

![Figure 9. Latent effectiveness comparison of coated wheels with various desiccants for (a) dehumidification (adsorption) and (b) regeneration (desorption). Results for SP15, HAS15, and SG13 are adopted from ref 30. SP-CPB0.5 (surfactant-coated starch particles), SP15 (native starch particles), HAS15 (high amylose starch), and SG13 (silica gel).](image3)
in Table 1, the SP-CPB0.5-coated exchanger had the lowest desiccant/matrix mass ratio among other coated sheets reported, despite the observation that the SP-CPB0.5 system outperformed the HAS15- and SG13-coated exchangers. In general, the latent effectiveness of the SP-CPB0.5-coated exchanger was between 5 and 30% higher, where the difference depends on the wheel angular speed, as compared with the SG13- or HAS15-coated exchangers. As well, surfactant coating onto the starch particles did not compromise the performance, as compared with the SP15 (native starch) coated exchanger. Therefore, SP-CPB0.5 represents a notable advancement in desiccant design for sustainable energy wheel fabrication. However, further research is needed to investigate the performance of SP-CPB0.5 at a wider range of operating conditions to assess its durability over a longer life cycle of operation.

**CONCLUSIONS**

This study reports on the water vapor adsorption−desorption process of starch particles (SPs) from the seed of the Prairie carnation that was surface modified with cetylpyridinium...
bromide (CPB) surfactant using a facile adsorption method. The SP-CPB0.5 sample (where the CPB loading concentration was maintained at 0.5 mM) denotes a CPB-coated SPs that was studied herein due to its unique surface/textural and water uptake properties. The SP-CPB0.5 sample was characterized using XPS, where various CPB doping levels provide evidence of favorable surface interaction of CPB with the surface of SPs. The morphology of SP-CPB0.5 was further characterized using TEM, which showed alteration of the layer-by-layer granule structure of pristine SPs, where agglomeration of the CPB-coated SPs is understood due to hydrophobic effects arising from CPB doping of SPs. Water vapor adsorption—desorption isotherms of pristine SPs and SP-CPB0.5 samples were compared for SPs coated with greater (5.0 mM) CPB loading (SP-CPB5.0). The trends in the relative water vapor uptake capacity at 25 °C at a fixed relative partial pressure (P/P₀ = 0.95) adopt the following order: SP-CPB5.0 (36.5%) ≥ SP-CPB0.5 (36.0%) > SPs (33.5%). The trend reveals a greater water uptake capacity of CPB-coated SPs over native SPs, in agreement with the creation of favorable pore and surface sites with enhanced vapor permeability SPs with surface-bound CPB. The single-step and steady-state cyclic experiments were conducted using a SP-CPB0.5-coated small-scale exchanger, where a comparison of native SPs revealed notable differences relative to high amylase starch (HAS₁₁₅) and silica gel (SG₁₁₃) desiccants. The latent effectiveness obtained from both the transient and steady-state methods for the adsorption (dehumidification) cycle are in good agreement within the experimental uncertainty limits (65.4 ± 2 and 64.6 ± 2%) for the cyclic and single-step experiments, respectively. The estimated latent effectiveness for the CPB-modified (SP-CPB0.5) and pristine SPs-coated exchanger was comparable and exceeded those of SG₁₁₃ and HAS₁₁₅-coated exchangers. Whereas the CPB surface coating onto SPs serves to improve the temporal longevity of the desiccant properties of SPs, the CPB coating of SPs does not compromise the moisture transfer (latent) capability relative to native SPs (without CPB coating). Thus, surfactant-coated starch particles (SP-CPB0.5) may serve as an alternative desiccant material for energy wheel applications with improvements to the wheel performance longevity of the desiccant at moderate temperature and humidity conditions, as compared with native starch desiccants.

## EXPERIMENTAL SECTION

### Materials

The granular SPs from the seed of the local Prairie carnation plant were supplied by Canadian Carnation BioProducts Inc. Cetylpyridinium bromide (CPB) was purchased from Sigma Aldrich (ON, Canada). Aluminum sheets (Al-3003, thickness of 0.62 ± 0.05 mm) were purchased from McMaster-Carr Supply Company.

### Preparation of CPB-Coated Starch Particles

The preparation of the CPB-coated starch particles (SP-CPBX; where X represent the loading concentration of the CPB surfactant) was adopted from a previous report. The SP-CPB0.5 sample was chosen for this study based on its unique spherical morphology and optimum water uptake and swelling properties as characterized in the previous study. The SPs with particle sizes less than 125 μm were screened with 120 mesh US standard sieve and then applied on the surface of a thin (0.035 mm) adhesive-aluminum tape attached to aluminum plates. Finally, any excess desiccants on the substrate surface were removed by blowing pressurized air to the desiccant-coated exchanger sheets. Sieving method does not use any external adhesives and the desiccant material directly adheres to the adhesive aluminum tape and thus the sieved desiccant particles were applied in a uniform monolayer on the Al substrate as described in a previous study. Upon completion of the coating, the SP-coated Al plates were left to dry in open air for 24 h, followed by application of a thin, uniform, and even layer of a 0.5 mM aqueous solution of CPB surfactant through a hand-held spray nebulizer. The CPB-modified SP-coated (SP-CPB0.5) Al plates were then dried in open air for 24 h, where the coating was observed to retain its original uniformity without any visible bulging or perforation. A small-scale parallel-plate exchanger (10 cm × 10 cm × 20 cm) was assembled using 16 Al sheets coated with SP-CPB0.5, as shown in Figure 10Bc. Table 1 compares details of the average mass (g) and mass per unit plate area (g/cm²) of the SP-CPB0.5-coated small-scale exchanger with previous reports on desiccant-coated exchangers.

### Transmission Electron Microscope (TEM)

TEM images of the SPs and SP-CPB0.5 samples were obtained using a Hitachi TEM system. The voltage of the system was set at 80.0 kV. SPs and SP-CPB0.5 samples were dispersed in methanol prior to deposition onto a carbon-coated copper TEM grid.

### X-Ray Photoelectron Spectroscopy (XPS)

XPS measurements were collected using a Kratos (Manchester, U.K.) AXIS Supra system equipped with a 500 mm Rowland circle monochromated, Al K-Alpha (1486.6 eV) source, and combined hemispherical analyzer and spherical mirror analyzer. A spot size with a hybrid slot (300 × 700 μm) was used. All survey scan spectra were collected in the binding energy range of 0–1200 eV with 1 eV steps and a pass energy of 160 eV. High-resolution scans of 4 regions were also collected using 0.05 eV steps with a pass energy of 20 eV. An accelerating voltage of 15 keV and an emission current of 15 mA were used for the analysis.

### Water Vapor Adsorption

The vapor adsorption analysis of the SPs and CPB-coated SPs was established using an intelligent gravimetric analyzer system (IGA-002) supplied by Hiden Isochema Ltd. (Warrington, United Kingdom). IGA-002 is equipped with a sensitive microbalance with a resolution of 0.1 μg and an uncertainty of ±1 μg. The sample holder is housed within a stainless steel reactor to create ultrahigh-vacuum conditions and eliminate changes in the external environment. In this vapor adsorption experiment, ca. 30–35 mg of solid SP samples were loaded in a stainless steel sample holder and placed in a reactor chamber. The desired temperature inside the reactor was precisely controlled using a water bath with the accuracy of ±0.1°C. Prior to the start of the isotherm measurements, the samples were thoroughly degassed and dried at 70°C under vacuum (≈10⁻⁵ mbar) for 6 h and held isothermally at 25°C. The adsorption isotherm measurements were acquired at 25°C for different pressure set points in the relative pressure (P/P₀) range of 0–1 with about 0.16 increments.

### Experimental Test Facility

In this study, two experimental approaches were used to determine the latent effectiveness of the CPB-coated SPs exchangers. Design and fabrication of the experimental test facility has been addressed
The schematic and photographs of the test facility are shown in Figure 10A,B, respectively. Figure 10A shows a schematic of the test facility consisting of two main sections: (I) air stream preconditioning section and (II) test section.

The preconditioning section consists of a supply air system where a compressor is in line with a dehumidifier. The air stream conditions are controlled by the humidifier, mass flow controllers, and heaters. The air stream temperature and relative humidity (RH) can be changed to simulate different air stream operating conditions. In the test section (Figure 10A), the air ducts 1 and 2 are the inlet streams to the test exchanger and are used for the counter-flow air supply to the exchanger. Flow straighteners are also placed in each air stream before the exchanger inlets to provide a uniform velocity profile at the inlet of the exchanger.

Thermocouples (6 in each duct) and RH sensors (4 in each duct) are placed upstream and downstream of the exchanger to measure the temperatures and relative humidity of air streams. RH and temperature of the outlet and inlet air streams were recorded with sampling frequency intervals of 1 s. A cycle generator unit, consisting of two linear slide actuators, moves the exchanger between the two air streams, and consequently subjects the exchanger to continuous dehumidification (adsorption) and regeneration (desorption) cycles. This allows us to convert the rotation of wheel to a linear motion and therefore simulate the actual operating conditions of a wheel. A signal-board microcontroller was used to control the actuators for various cycle frequencies, with frequency as high as 1 Hz (1000 ms period).

**Single-Step and Cyclic Experiments.** The experimental test facility, shown in Figure 10, was used to subject the SP-CPB0.5-coated plates within the small-scale exchanger to a single-step change (termed single-step experiment) or a series (cycles) of step changes (termed cyclic experiment) in the inlet humidity. The condition of dry and humid air streams for the single-step and cyclic tests are the same as those of the previously reported literature listed in Table 1. In the single-step experiment, the preconditioning of the test section was initially carried out to achieve steady-state conditions for the test facility. Then, a sudden step change in humidity was made in less than 1 s. The test was run until the outlet air stream reached a steady-state condition. In a single-step change experiment, the steady-state is defined as the condition under which variations of temperature and RH of a single air stream remain within the sensor uncertainty for a period of 5 min. In the cyclic experiment, the exchanger was exposed to a series of continuous adsorption/desorption cycles, and the test was run until the outlet air streams and air ducts 3 and 4 (Figure 10A) reached a steady-state condition. The exchanger is also considered to be in a dynamic steady state when the difference between average effectiveness of two consecutive cycles is less than 1%, which is below the uncertainty limits. To compare the results with those of a previous report, 120 s was used as a period for one cycle, which is equivalent to 0.5 rpm.

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