Preparation and Properties of Foamed Cellulose-Polymer Microsphere Hybrid Materials for Sound Absorption

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Sustainability and eco-efficiency are presently directing the development of the next generation of acoustic materials. In this work, foamed cellulose-polymer microsphere (PM) hybrid materials, having sound-absorbing capability, were prepared by incorporating the PMs into cellulose fibers by dehydration and foaming processes. The evolution in morphology of PMs during foaming process was investigated for different heating temperatures. The beating process disintegrated the microscopic cellulose fiber into the smaller fibers, which connected the PMs by a unique fibrous network. The influences of foaming temperature, PM content, and total areal density on the sound absorbing property of composites were studied. The results showed that incorporating the acoustic unit of elastic PMs into the porous cellulose fiber-based network significantly improved the sound absorbing ability of the composites. The sound-absorbing hybrid materials appear to be a promising alternative to non-degradable organic or inorganic acoustic composites, being economical, simple, and eco-friendly.

Keywords: Cellulose fibers; Acoustic materials; Foaming process; Beating process; Sound absorption coefficient

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INTRODUCTION

Currently, air, solid, and water pollution are considered potentially most dangerous, whereas there has been a lack of awareness concerning noise pollution (Okada and Inaba 1990). Noise pollution, especially low-frequency noise (20 to 100 Hz), is harmful to the human body, because of the resonance of audio frequency and the internal constituents, which can produce damage to internal organs (Berglund *et al*. 1996). Noise pollution is also one of the most important environmental problems and is harmful to human health, as is exemplified by hearing loss and body lesions (Stansfeld and Matheson 2003). The use of sound-absorbing hybrid materials is widely considered to be an ideal approach for reducing noise pollution because of their unique porous structures, remarkable sound absorbing properties, and easy installation. In recent years, many types of sound-absorbing hybrid materials have been developed, and they are primarily composed of synthetic plastics or inorganic absorbers and metal-based microperforated panel absorbers. These sound-absorbing hybrid materials have the disadvantages of non-degradability and scarcity or increased expense of key resources, although there have been much wider applications (Egab *et al.* 2014).

Alternatively, the production of new sound-absorbing hybrid materials starting from the renewable resource cellulose can be an efficient strategy on a large scale. The use of cellulosic materials can provide biodegradability at low cost, combined with remarkable sound absorbing properties (Yeon *et al.* 2014). In 2008, our research group reported the preparation of biocomposites composed of biodegradable cellulose fibers and polyurethane adhesive (MPU-20). These composites exhibit promising sound-absorption properties and have higher absorption coefficients over a wide frequency range (250 to 7000 Hz) (Liu *et al.* 2008). In 2011, our group also fabricated a new green sound-absorbing hybrid material *via* pulp fibrous models, in which the natural pulp fibers have appreciable sound-absorbing properties because of their technical advantages, including being porous, flexible, and easy fibrillated, compared with inorganic glass- and aluminum-silicate fibers (Liu *et al.* 2012). In 2012, the authors' group further manufactured gradient acoustic pulp/granular particle biocomposites in which the granular particles included rubber particles, white carbon black, and ceramic microspheres. These structures were demonstrated to improve the sound absorption coefficients (Liu *et al.* 2013). The elastic hollow polymer microsphere (PM) was reported to have sound absorption superiority in a low sound frequency range (250 Hz to 1500 Hz) but not at high sound frequencies ($\geqslant$1500 Hz) (Lauriks *et al.* 1989; Hong 2004; Zhou *et al.* 2004). Moreover, the cellulose fiber-based network exhibits outstanding sound absorbing property at high sound frequencies ($\geqslant$1000 Hz) but not at low sound frequencies. The sound absorption phenomena inside fibrous and porous materials arise primarily because of energy dissipation involving viscosity and thermal conductivity. Once the sound wave reaches the acoustic materials, part of the wave is reflected, while the rest enters the materials. The main absorbing mechanism is based on the porous structure of fibers and the relaxation of elastic polymer microspheres (Jarzynski 1990). For porous fibers, including glass wool, rock wool, slag wool, glass filaments, and microfibers, sound waves get into the material along the pores and further convert the energy of sound into heat by friction. Polymer microsphere materials exhibit strong sound-absorbing properties at low sound frequencies but weak capability at high sound frequencies. Composites based on a fibrous matrix and granular materials are used to achieve sound absorption effects over an extensive sound frequency. However, scientific knowledge about processing elastic hollow PMs into a macroscopic cellulose fiber-based network is still scarce and is a topic of extensive current research (Li *et al.* 2008).

This study reports the preparation of a new foamed cellulose-polymer microsphere materials combining elastic hollow PMs and cellulose fibers by typical dehydration and foaming technologies as well as the changes during the process. The unique porous structure provides composites having promising sound absorbing properties at certain sound frequencies.

EXPERIMENTAL

Materials

A commercial bleached softwood kraft pulp used as the raw cellulose source material was purchased from Guangzhou Chenhui Paper Co., Ltd. (China). The raw cellulose fibers have an initial 10 wt% moisture content and beating degree of 14 °SR. The PM provided by Matsumoto Yushi Seiyaku Co., Ltd. (Japan) is an elastic thermal expansion microcapsule, which is available in the following four types: F-36D, F-36, F-48,

and F-50. The dispersant poly-ethylene oxide (PEO) with a molecular weight of400 million was provided by Shanghai League Victory Chemical Co., Ltd. (China)Sodium carboxymethylcellulose (CMC), provided by Beijing Dream YiMei Biotechnology Co., Ltd.(China), was used as a natural adhesive between PMs and cellulose fibers.

Pretreatment of Raw Materials

The evolution in morphology of PMs during the foaming process was investigated by stereoscopic microscope (SZX12 model, Shanghai Optical Instrument Factory, China). The foamed PM samples at temperatures ranging from 90 to 140 °C were drawn by blood capillary (Huaxi Medical University Instrument Factory, China) (100 mm in length, 0.5 mm in diameter), followed by placing them onto a microslide for observation. The raw cellulose fibers were pretreated by a beating process with a PFI mill (Hamjern Maskin 621, Dongguan International Material Tester, China).

The beating degree of pulp suspensions was determined, and the pulp fiber characteristics were analyzed as a function of different beating degrees using a Kajaani FS300 Fiber Analyzer (Metso Automation Inc., Finland). The pulp fiber characteristics (such as fiber length, width, crimp index, and kink index) were controlled through the PFI refining process. Morphological properties including fiber fines content (TAPPI T271 2012) and fiber kink and crimp index values were determined with the Kajaani FS300 fiber analyzer.

Preparation of Foamed Sound Absorbing Hybrid Materials

A set amount of pulp sample was soaked in water at room temperature for 24 h and subsequently disintegrated by a Deflaker (Dongguan HengkeInstrument Factory, China) followed by PFI beating with different processing times and at rotational speeds of 5000 to 14,000 rpm to obtain various pulp-drainage levels.

The pretreated pulp slurry was diluted to a 0.2 wt% consistency and mixed with 30 to 100 wt% PM suspensions along with1.0 wt% PEO and 1.0 wt% CMC. The above mixture was efficiently mixed and poured into a Model-1600 Econo-space Automated Sheet Formation System (RéalisationsAustrales Inc., Canada) after removal of water using a vacuum or hydraulic pressure molding. After completing the formation, the samples were dried at temperatures ranging from 100 to170°C for the foaming process, using the forced air-drying method. The effects of foaming temperature on the thickness of composites were studied.

Thermal Properties Analysis

The thermal properties of PMs were analyzed by differential scanning calorimetry (DSC-2010, TAQ200 Instruments Inc., New Castle, DE). A mechanical refrigeration system (RCS) was used for all measurements, including a sealed empty pan as a reference using nitrogen gas as the flushing agent over the head.

The temperature and melting calibrations were performed with indium [mp=156.6 °C and $\triangle H_m$=28.5 J/g]. A total of 8 to 9 mg of the sample was placed in hermetically sealed aluminum pans and heated (80 to 300 °C) at a heat flow rate of 10°C/min under constant purging of nitrogen (30 mL/min).

Scanning Electron Microscopy Analysis

Samples of air-dried pulp fibers were fixed to a metal specimen holder using double-sided sticky tape and were coated with gold using a vacuum sputter-coater (Zhong Xian Heng Ye Instrument Factory, China). The cross-section profiles of air-dried pulp fibers and PMs were observed using a field-emission scanning electron microscope (Zeiss LEO 1530VP, Germany).

Sound-Absorption Analysis

Sound-absorption coefficients (Hirosawa *et al*. 2009) of the composites samples were obtained experimentally using a sound absorbing analyzer (AWA6122 model, Aihua Instrument Factory, China) equipped with two tubes as exhibited in Figs. 1a and 1b. The L tube type of 100-mm diameter standing wave tube was for low-frequency (⩽1500 Hz) testing, while the S type 30-mm diameter standing wave tube was for high-frequency (⩾ 1500Hz) tests. The procedure of measuring sound absorption of the samples was that they are first prepared with two different diameters of 30 mm used for high sound frequencies (*i.e*., 800 to 7000 Hz) and another 100 mm used for low frequencies (*i.e*., 90 to 1800 Hz). The sound absorption measurement was ready according to Fig. 1a, and the data collection system was connected to a computer for automatically processing data. In this process, the sample must contact the sample tube very well to prevent the leakage of acoustic wave. These tests were carried out using ISO E10534-2 method. (Geneva, CP, Switzerland, 1998) The effects of basis weight, thickness, beating degree, and PM content of the samples on the sound-absorbing properties were also investigated.

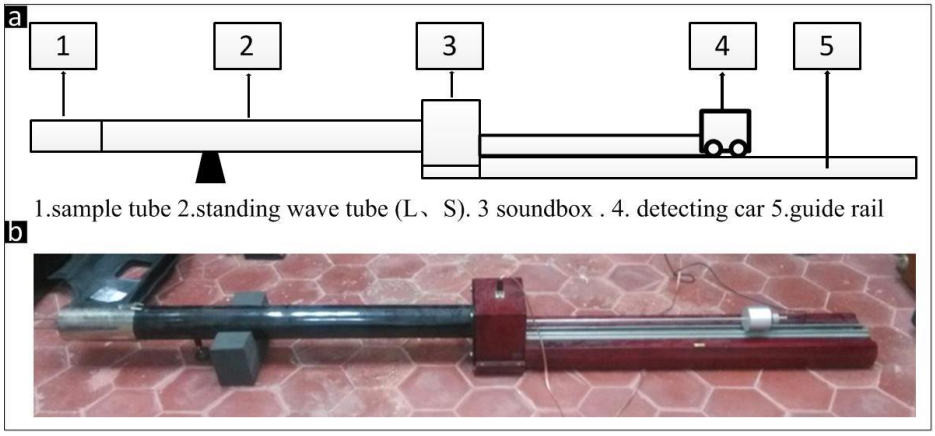

Fig. 1. (a) Schematic illustrating the working mechanism of acoustic tests. L standing wave tube is for low-frequency (≤1500Hz) test. S standing wave tube is for high-frequency (≥1500 Hz) test; (b) picture of AWA6122 electroacoustic test system.

RESULTS AND DISCUSSION

Foaming Process Analysis

Polymer microspheres, as discussed in the article, are elastic polymers that contain an alkane-based compound liquid with low boiling point. The polymer shell is highly elastic and designed to seal the alkane-based compound (Gershtein 1973). Figure 2a indicates that the gasification of alkane-based compound liquids in heating process expanded the microspheres' size to a large degree. A hollow structure sealed with an elastic

polymer shell appears during the foaming process. Furthermore, PMs do not shrink to their original states when free of external heating process, nor does the hydrocarbon gas generated by PMs condense into liquid droplets. More importantly, compared with ordinary chemical decomposition reactions, these processes of transformation are stable and irreversible (Sohn and Szekely 1972).

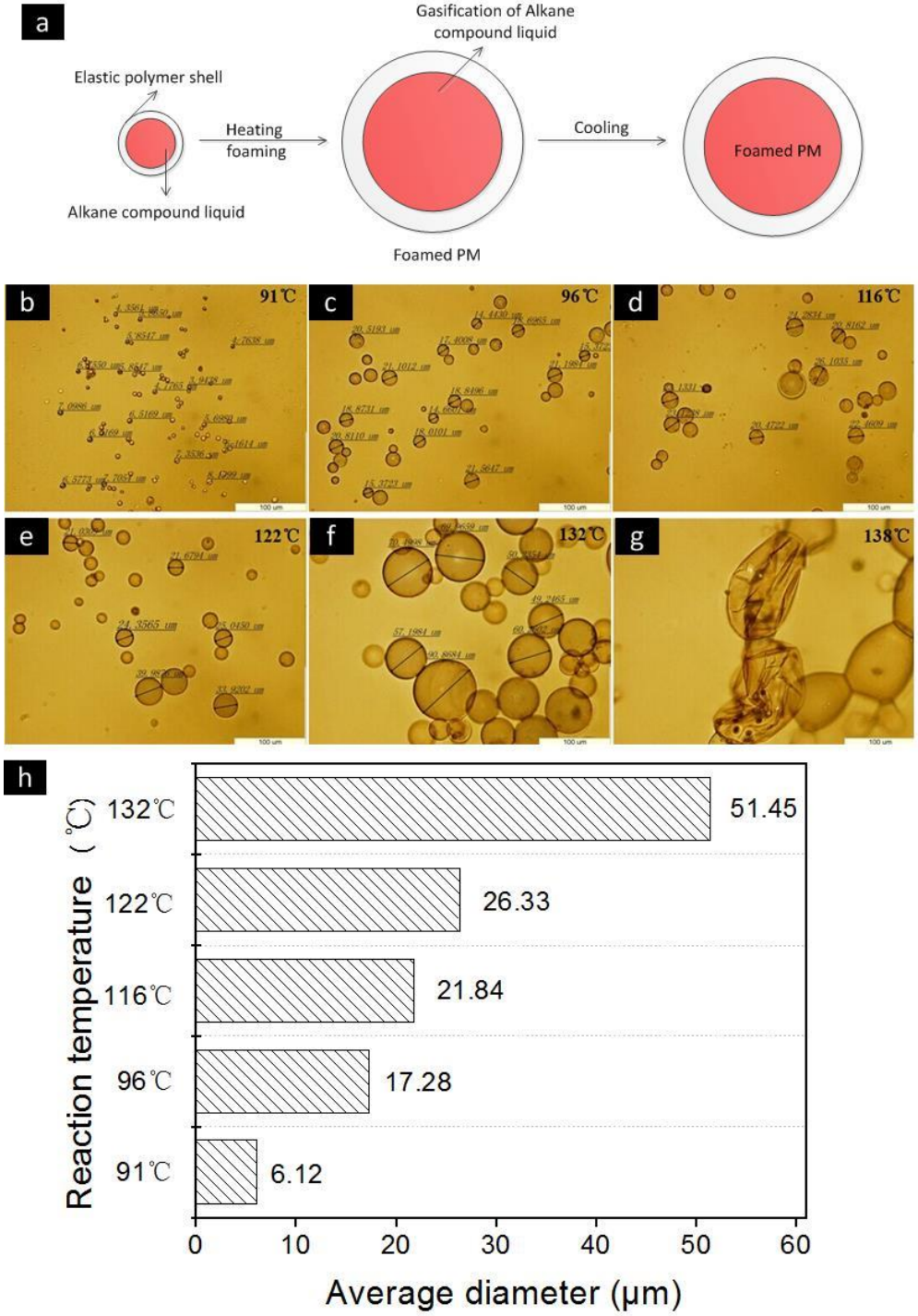

Fig. 2. (a) Schematic illustration of the foaming process of the PMs. The optical microscope pictures of PMs (*e.g.*, F-36D model) were acquired at heating temperatures of (b) 91, (c) 96, (d) 116, (e) 122, (f) 132,and (g) 138 °C; (h) the average diameter of F-36D at different temperatures.

It is to be noted especially that the generated hydrocarbon gas, with a low boiling point, is not affected by a temperature decrease and will not condense into a liquid. The imbalanced pressure difference inside and outside the shell would result in the hollow structure of PMs being compressed and destroyed if these released gases condensed into a liquid. As shown in Figs. 2b to h, inside the PMs, increasing environmental temperature vaporizes the liquid alkane compound to gaseous form with different dimensions. Nevertheless, the PMs have promising compression resistance upon the gas pressure produced by vaporization of inner liquid without fracture after the foaming process (Chen *et al.* 2008). Moreover, the polymer shell is able to expand easily in the case of heating, but its dimensions are maintained unchanged after a cooling process. However, the high pressure inside PMs can destroy the polymeric shell when sharply increasing the heating temperature above 138 °C (see Fig. 2g).

Figure 3 shows differential scanning calorimetry (DSC) results for different kinds of PMs. The DSC figures, indicating the approximate reaction temperature of all four kinds of PMs, paved the way to determine a more accurate reaction temperature. For the case described in Fig. 3a, the DSC data are shown as drastically fluctuating at 112.44, 123.5, and 135.32 °C, indicating that more experiments have to be conducted at 110, 120, and 130 °C. In total, four kinds of PMs (F-36D, F-36, F-48, F-50) were investigated to explore the influence of heating temperature on the PMs and determine an accurate reaction temperature.

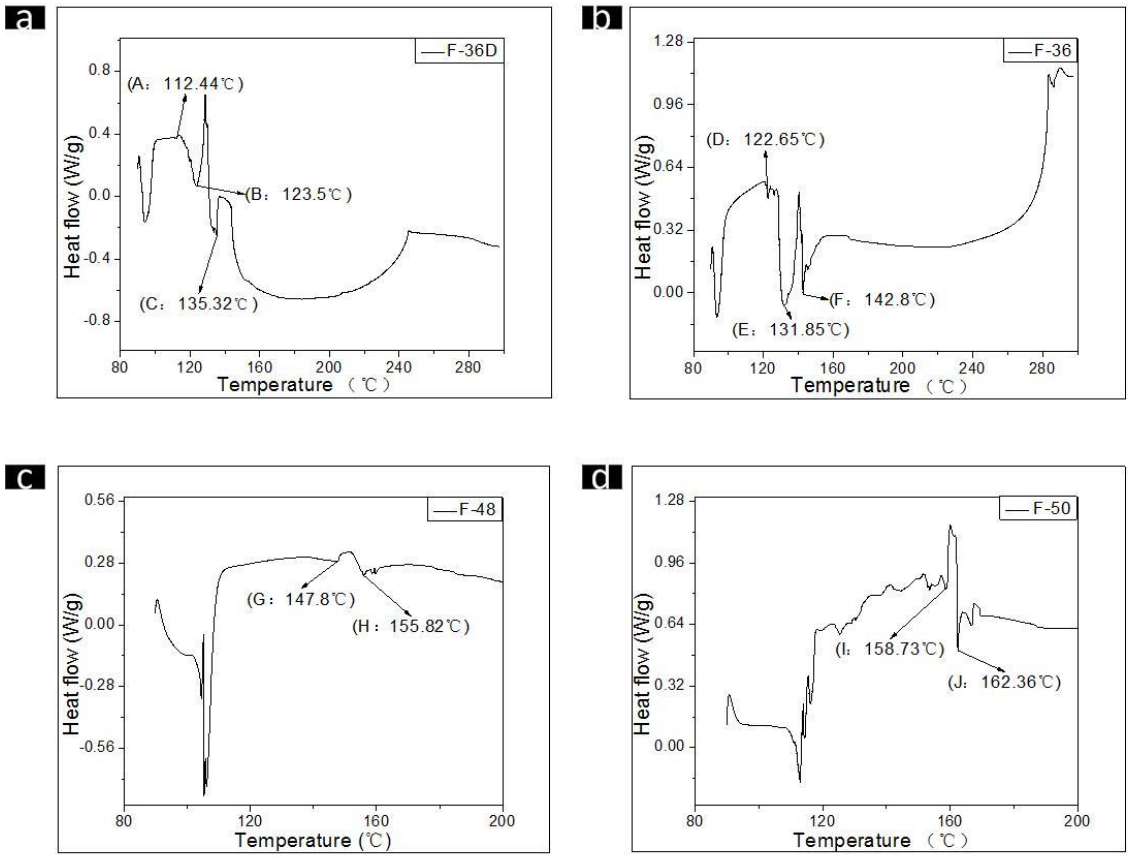

Fig. 3. Differential scanning calorimetry (DSC) results for PMs:(a) F-36D, (b) F-36, (c) F-48, and (d) F-50

The changes in thickness of PMs at different temperatures (Lee and Chang 2004), PM content, and basis weight are shown in Figs. 4a-d, Fig. 4e, and Fig. 4f. Figure 4 indicates that the proper reaction temperatures for F-36D, F-36, F-48, F-50, were 110, 130, 160, and 170 °C, respectively. Compared with other PMs, F-36D exhibited the highest rate of thickness growth, whereby the thickness change rate increased to 88.83% at 110°C. This can be explained by the difference in boiling point, as each kind of PM had a unique boiling point and effect of expansion. Moreover, it is more efficient (as can be seen from Fig. 3f, where 500 g/m^2 performed better than 1000, 1500, and 2000 g/m^2) to choose the basis weight of 500 g/m^2, for which the thickness change rate increased to 74.8%. In general, the porous structure plays an important role in the properties of sound absorption as well as the strength of a material (Zhang and Chen 2009).

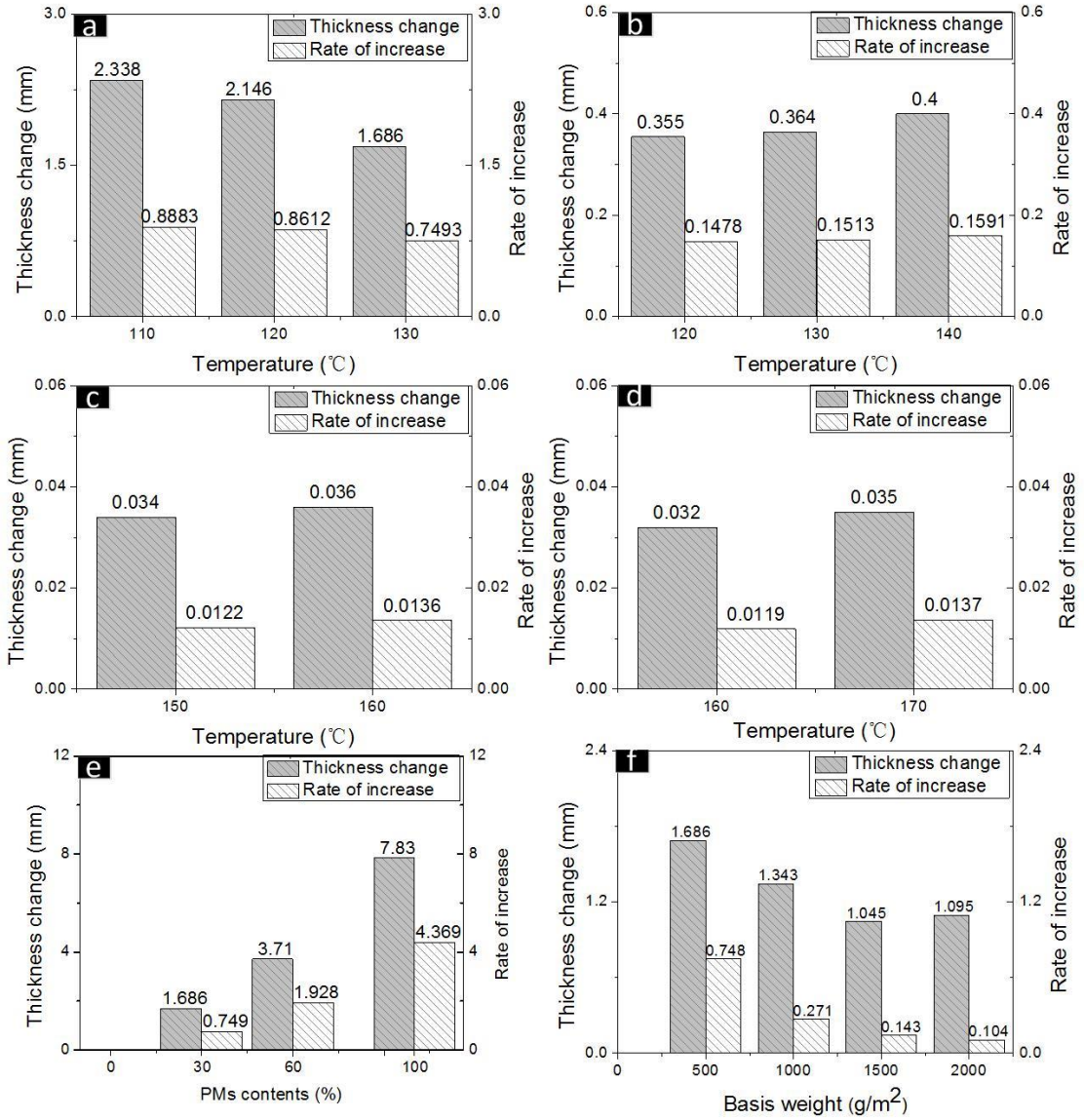

Fig. 4. Rate of increase and thickness change at (a) 110, 120, and130 °C (F-36D), (b) 120, 130, and140 °C (F-36), (c) 150 and160°C (F-48), and (d) 160 and170 °C (F-50); (e) rate of increase and thickness change of F-36D with PM contents of 0, 30%, 60%, and100%; (f) rate of increase and thickness change of F-36D with basis weights of 500, 1000, 1500, and 2000 g/m^2

Relationship between Cellulose Morphology and Beating Degree

Figure 5 shows the effects of beating degree on the fines content, kink index, and crimp index. Compared with the raw pulp fibers, the kink index and crimp index decreased with increasing beating degree, as measured by the Schopper-Riegler method (Li and Xu 2011), from 14 °SR to 68 °SR, but the fines content increased as well (Attenborough 1971; Na *et al.* 2007; Beg and Pickering 2008). As for the fines content test, measurements controlled by computer were based on the direction of polarized light. Pulp fibers were squeezed and kneaded repeatedly by rectangular metal edges on refiner plates during the PFI-beating process, resulting in crimping or kinking of pulp fibers (Gao *et al.* 2015). Thus, many small pores were filled by cellulosic fine particles, leading to the decrease in sound absorbing properties. The same trend occurred with decreasing crimp index and kink index (Shatalov and Pereira 2002). Figure 6 shows scanning electron microscopy (SEM) images of the original cellulose fiber-based materials without PMs and the foamed cellulose/PM materials. With PMs present, the cellulose/PM composites have strong sound absorbing properties (as shown in Fig. 6b), there exists many foamed PM. These small microspheres can perform well in sound absorption not only in theory, but also as shown by Fig.7d.).

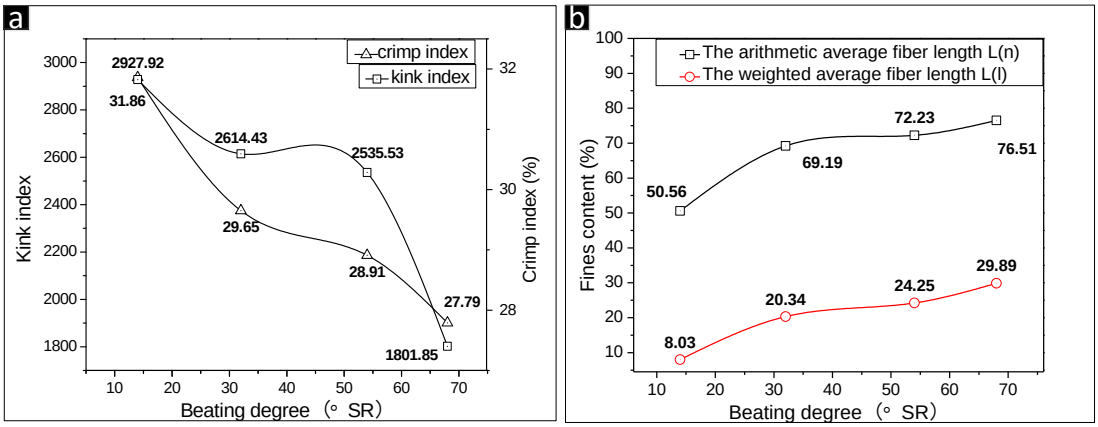

Fig. 5. (a) Kink index and crimp index at different beating degrees;(b) fines content at different beating degrees

Fig. 6. SEM images of (a) the original cellulose fiber-based materials without PMs and (b) the foamed cellulose/PM materials

Sound Absorption by Cellulose-Polymer Hybrid Materials

In general, every kind of PM has a corresponding reaction temperature. Usually, an absorption coefficient over 0.2 can justify use of the term sound-absorbing material, and a value over 0.5 means that it can be called a very good sound-absorbing material. As shown in Fig. 7a, 110 °C is perfect for F-36D, especially in a high frequency range (>1500 Hz), although not good at a low frequency range (<1500 Hz). Our materials perform well especially over 3000 Hz, and the sound absorption coefficient was able to exceed 0.60 at 5000 Hz, which is better than 120°C and 130 °C. To achieve a good sound absorption coefficient, it is efficient to add 30% PMs, as shown in Fig. 7b. On the other hand, Fig.7c shows the sound absorption coefficients at different basis weight for wood fiber biocomposites. Composites with a basis weight controlled at 2000 g/m^2 perform well, especially at a high frequency range (>1500 Hz). Figure 4f indicates that thickness change rate of 500 g/m^2 was better than that for 1000, 1500, and 2000 g/m^2 specimens. The basis weight of 2000 g/m^2 exhibited a better absorption coefficient than 500 g/m^2, but it also required more cellulose and PMs.

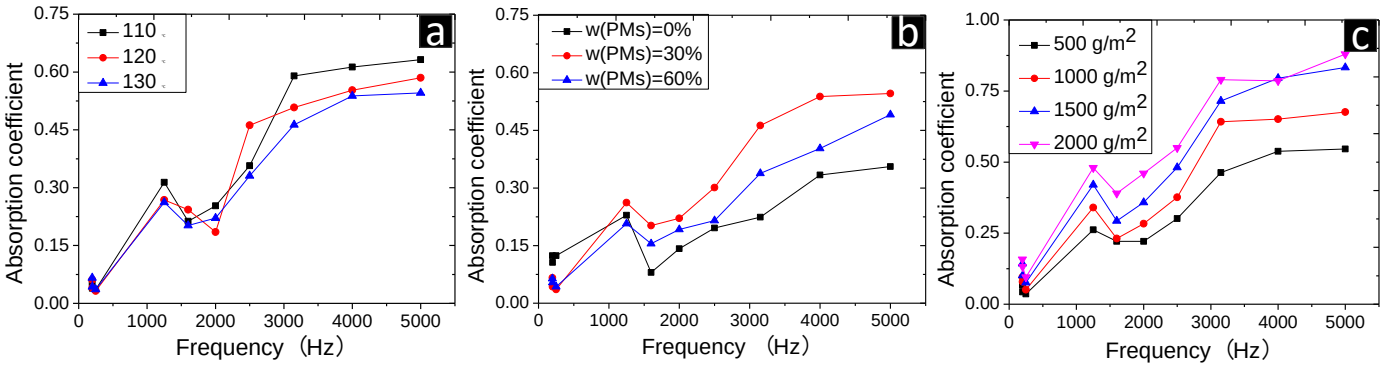

Fig. 7. Variation of the absorption coefficient with frequency at various (a) temperatures, (b) PM contents, and (c) basis weights.

CONCLUSIONS

1. The preparation of cellulose-polymer sound-absorbing hybrid materials is reported in this work. Each kind of polymer microsphere (PM) responds to an optimal reaction temperature. Among these PMs, F-36D performed best at 110 °C, with a thickness change rate that increased by 88.3%. Moreover, it is more efficient to choose the basis weight of 500g/m^2 because of the highest thickness change rate compare to other basis weight, which has increased by 74.8%.
2. With increasing beating degree, the pulp fibers' crimp index and kink index decreased gradually, but fiber fines content increased. To obtain good sound absorption properties, the beating degree should be set at 14 °SR. In fact, the sound absorption was excellent, especially at high frequencies (≥1500Hz), when 30% PMs were added, the sound absorption coefficients can reach to 0.58. These composites can be regarded as ideal, since an absorption coefficient over 0.2 means that the material can be called sound-absorbing. An absorption coefficient over 0.5 means that the specimen can be called very good sound-absorbing material. The material prepared in this work performed well, especially over 3000Hz), meaning that it was a sound absorbing hybrid material

suitable for high frequencies, and this study presents arguably the most versatile as well as easily scalable method to generate these remarkable sound absorbing hybrid materials.

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