# Characterization of Soy Protein–Celery Composite Paper Sheet: Rheological Behavior, Mechanical, and Heat-Sealing Properties

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**ABSTRACT:** The rheological properties of soy protein spray-coating dispersions were investigated as a function of concentration (10–13.75%) and temperature (30–60°C). The dispersions showed a shear-thinning behavior. With the increase in concentration, the consistency coefficient ($\kappa_H$) values increased. An increasing in temperature led to an increasing flow behavior index ($n_H$) and a decreasing $\kappa_H$. However, Cox–Merz rule was not applicable to the coating dispersions. Soy protein–celery composite paper sheet was prepared by spraying soy protein on the surface of the celery paper sheet. The cohesion of soy protein and celery fiber resulted in sealing of celery paper. The seal strength increased with the increase in soy protein concentration. The seal strength increased from 114.850 to 160.560 N/m, when the soy protein concentration increased from 10 to 13.75%. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

**Key words:** rheological properties; soy protein; celery paper; heat-sealing properties

## INTRODUCTION

Because of the limitation of resources and environmental unfriendliness for using nondegradable synthetic products as food packaging materials, the development of edible films and biodegradable packaging materials from nature recourses, especially agriculture products, has received a great deal of current interests.[1–6]

The properties of edible films are dependent on the film-forming materials used.[7] Soy protein-based edible films have received high attention owing to their excellent film-forming abilities, good biodegradable performance, low cost, and barrier properties against oxygen permeation at low relative humidity (RH) conditions.[8] However, the unsatisfactory mechanical properties, coupled with strong moisture and temperature sensitivity during service life, have limited the use of these materials.[9] Vegetable paper, which is made from vegetables, is a potential new type of edible packaging material. However, poor heat-sealing performance has limited the application of vegetable paper in the packaging field.

Several studies concerned with the rheological characteristics of soy protein suspension individually,[10,11] soy protein gels,[12,13] or mixed with food system.[14–16] Nevertheless, no attempts have been made so far to study the rheological properties of soy protein spray-coating dispersions. Furthermore, no data were available on vegetable paper heat-sealing properties. Considering these problems, soy protein–celery composite paper was prepared by spraying soy protein on surface of the celery paper sheet. The objective of this study was to (a) investigate the rheological properties (stationary and dynamic) of the soy protein spray-coating dispersions and (b) obtain soy protein–celery composite paper and evaluate their mechanical performance and heat seal ability.

## EXPERIMENTAL

### Materials

Celery was purchased from Tianjin local markets, Tanjin, China; Soy protein powder (Tangshan Wang Li Da Co., Hebei Province, China), glycerol, sodium carboxyl methyl cellulose (CMC, pH = 9.2), and polycarboxylic salt additives were provided by Tianjin Chemical Co., Tianjin, China.

### Preparation of soy protein dispersion

Four concentration levels of soy protein dispersions (Table I) were obtained by dispersing soy protein powder in distilled water under stirring with a

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**TABLE I**
**Composition of Investigated Soy Protein Spray-Coating Dispersions in Wt %**

| Soy protein powder | Polycarboxylic salt | CMC | Glycerol |
|---|---|---|---|
| 10 | 12.5 | 12.5 | 10 |
| 11.25 | 12.5 | 12.5 | 10 |
| 12.5 | 12.5 | 12.5 | 10 |
| 13.75 | 12.5 | 12.5 | 10 |

mechanical agitator at 500 rpm and 80°C in a water bath for 60 min. CMC (1%, w/w), glycerol, and polycarboxylic salt (1%, w/w) were then added in turn and subjected to stirring for another 30 min. The mixtures were then poured into a beaker and set aside at room temperature.

### Preparation of celery paper and composite paper

The preparation of celery paper sheet was performed in a Fourdrinier papermaking machine designed by the Tianjin University of Commerce according to the patent of ZL200710060405.4.[17] The celery was washed with tap water to remove impurities and chipped into nominally 4 cm length, and then 200 kg celery were beaten in a beater for 100 min to form pulp. The pulp was poured into a large tub, transferred to the stuff chest where, and then diluted in a mixing chamber with more water to a consistency of about 0.5%. The diluted pulp passed from the stuff chest to the head box of the Fourdrinier to produce a uniform fiber suspension depositing on a wire mesh to drain. The celery fibers intermeshed and arranged themselves into a mat of paper. When the paper reached the end of the wire mesh, it was transferred to a felt blanket, which conveyed it through heating dryer to remove the excess moisture. In the process, the paper gets some glaze like coating also. The paper was then rolled and conditioned at room temperature.

Preparation of soy protein dispersion according to Table I, Process flow chart for the production of soy protein–celery composite paper sheet is presented in Figure 1.

### Rheological measurements

Rheological measurements were carried out using an advanced stress/strain controlled rheometer (Physica MCR 301, Anton Paar GmbH, Austria) equipped with a cone-plate geometry (50 mm of diameter, 2 cone angle, and 0.208 mm gap). For all the rheological tests, the temperature was fixed at the desired temperature (30, 40, 50, and 60°C), which was regulated by a circulating water bath and a peltier heating system. After loading, the sample was trimmed and left to rest for 3–5 min.[18]

### Steady shear measurements

Steady flow behavior of prepared soy protein dispersions was evaluated at strain-controlled mode; the amount of dispersions was appropriately 1.2 mL. The flow curves, shear stress versus shear rate, were plotted by increasing shear rate. Obtained data were fitted to Herschel–Bulkley's model using the equipment's Rheoplus/32V3.40 software.

Herschel–Bulkley's model:

$$\tau = \tau_{0H} + \kappa_H(\dot{\gamma})^{n_H} \quad (1)$$

where $\tau_{0H}$ is the yield stress (Pa), $\kappa_H$ is the consistency coefficient (Pa $s^n$), and $n_H$ is the flow behavior index for Herschel–Buckley's model.

#### Dependence of concentration

The concentration ($C$, %) effect at each temperature on apparent viscosity of soy protein dispersions, at a specific shear rate of 100 $s^{-1}$, can be described by power law [eq. (2)] and exponential [eq. (3)], functions[19,20]:

$$\eta_{100} = a_1 C^{b_1} \quad (2)$$

$$\eta_{100} = a_2 e^{b_2 \times C} \quad (3)$$

#### Dependently of temperature

The temperature dependency of apparent viscosity (at a shear rate of 100 $s^{-1}$) for each soy protein concentration was evaluated by applying the Arrhenius model.[19,21]

$$\eta_{100} = Ae^{\left(\frac{E_a}{RT}\right)} \quad (4)$$

where $A$ is the consistency related to structure and formulation (Pa s), $E_a$ is the activation energy (J/mol), and $R$ is the universal gas constant (J/mol k).

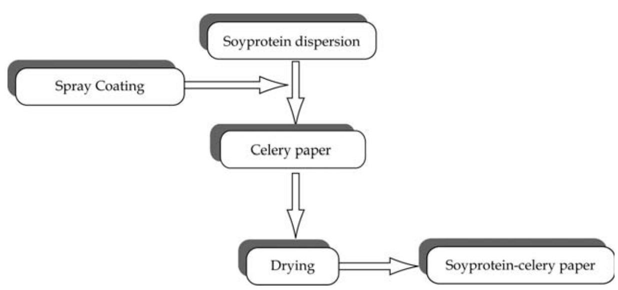


**Figure 1** Process flow chat for production of composite paper.

### Dynamic viscoelastic behaviors

Dynamic amplitude sweeps (0.01–100% strain) were performed under a constant frequency (10 rad/s), in order to determine the linear viscoelastic range, in which rheological properties were not strain or stress-dependent, and then frequency sweep tests (1–100 rad/s) were conducted for all samples. In oscillatory tests, samples were subjected to sinusoidal oscillating stress or strain with a frequency, and the elastic or storage modulus ($G'$) and the viscous or loss modulus ($G''$) and $\eta^*$ were determined as a function of frequency.[22]

### Composite paper characterization

#### Mechanical properties

All composite papers were conditioned at 23°C and 50% RH for 48 h in an environmental chamber, according to ASTM standard method D 882-01.[23] A texture analyzer (XLW-200N, Intelligent Electronic Tensile Tester, Jinan, China) was used to measure tensile strength (TS). The initial grip separation was set at 250 mm, and the crosshead speed was set at 25 mm/min. Ten specimens (300 mm × 25 mm) of each sample were tested.

#### Heat-sealing properties

Soy protein–celery composite paper-sealing strength was measured according to ASTM standard method F88-00.[24] Heat sealing apparatus (HSG-C, Switzerland) was used for soy protein–celery sealing treatment. The composite paper, which coated with soy protein dispersions, was overlapped for sealing. All the soy protein–celery composite paper samples were conditioned in a constant-temperature humidity chamber set at 23°C with 50% RH for 48 h to adjust the moisture content. The initial grip separation and the crosshead speed were set at 100 mm and 250 mm/min, respectively. Ten specimens (80 mm × 25 mm) for each paper sample were tested, and their mean values were reported.

## RESULTS AND DISCUSSION

### Steady shear properties

The rheological parameters of soy protein spray-coating dispersions obtained by applying the experimental shear stress–shear rate data to the Herschel–Bulkley's model as a function of temperature and concentration are given in Table II. All the samples had non-Newtonian shear-thinning behavior with values of flow behavior index ($n_H$) ranging from 0.56 to 0.79. Shear-thinning behavior is caused by a decrease in entanglement density and the alignment or arrangement of chain segments of polymers in the direction of applied shear stress.[25] The magnitudes of consistency coefficient ($\kappa_H$) values increased with the increase in concentration. Higher values of $\kappa_H$ indicated a more viscous nature owing to increase of soy protein concentration. Several previous works reported that the value of flow behavior index ($n$) changes with the concentration and is highly influenced by the molecular size; also, in general, when concentration or molecular weight increases, flow behavior index ($n$) decreases and $\kappa$ increases.[26–28] In this study, the magnitudes of $n_H$ were observed to increase with the increase in soy protein concentration, but decrease in high concentration (13.75%). This phenomenon indicated that this dispersion, at high concentration, has a high viscosity at low shear rates and strong shear-thinning characteristics.[26] It was also observed that increasing temperature led to higher $n_H$, while lower $\kappa_H$. The Herschel–Bulkley's model was adequate for describing the flow behavior of the soy protein dispersion, at higher temperature (50 and 60°C), where determination coefficients ($R^2$) were higher than 0.997. However, some negative yield stress values were obtained at lower temperature (30 and 40°C). This suggested that the Herschel–Bulkley's model was not so adequate for describing the flow behavior of soy protein dispersion (Table II).

**TABLE II**
**The Herschel–Bulkley Parameters for Soy Protein Spray-Coating Dispersions at Different Concentrations and Temperatures**

| Concentrations (%) | Temperature (°C) | $\tau_{0H}$ (Pa) | $\kappa_H$ (Pa s$^n$) | $n_H$ | $R^2$ |
|---|---|---|---|---|---|
| 10 | 30 | −0.37 | 0.68 | 0.60 | 0.997 |
| | 40 | −0.17 | 0.62 | 0.61 | 0.998 |
| | 50 | 0.11 | 0.33 | 0.71 | 0.999 |
| | 60 | 0.15 | 0.21 | 0.76 | 0.997 |
| 11.25 | 30 | −0.24 | 0.74 | 0.61 | 1.000 |
| | 40 | −0.22 | 0.43 | 0.68 | 0.999 |
| | 50 | 0.0089 | 0.32 | 0.75 | 0.997 |
| | 60 | 0.091 | 0.27 | 0.77 | 0.998 |
| 12.5 | 30 | −0.26 | 1.02 | 0.62 | 0.994 |
| | 40 | 0.068 | 0.57 | 0.70 | 0.994 |
| | 50 | 0.26 | 0.32 | 0.79 | 1.000 |
| | 60 | 0.27 | 0.31 | 0.79 | 1.000 |
| 13.75 | 30 | −0.84 | 1.56 | 0.56 | 0.997 |
| | 40 | −0.33 | 1.00 | 0.63 | 0.997 |
| | 50 | 0.051 | 0.67 | 0.70 | 0.998 |
| | 60 | 0.30 | 0.58 | 0.72 | 0.999 |

### Influence of soy protein concentration

For making direct comparisons among the several of soy protein dispersions, the apparent viscosity ($\eta_{100}$) at a specific shear rate of 100 s$^{-1}$ was used. The shear rate value was selected as it was associated

TABLE III
**Concentration Dependency of Apparent Viscosity of Soy Protein Spray-Coating Dispersions at Different Temperatures and Constant Shear Rate (100 $s^{-1}$)**

| Temperature (°C) | $a_1$ (mPa s/%) | $b_1$ | $R^2$ |
|---|---|---|---|
| (a) Power model | | | |
| 30 | 0.530 ± 0.120[a] | 2.260 ± 0.009[a,b] | 0.973 |
| 40 | 0.340 ± 0.009[b] | 2.390 ± 0.100[a] | 0.981 |
| 50 | 0.530 ± 0.063[a] | 2.130 ± 0.043[b] | 0.987 |
| 60 | 0.580 ± 0.170[a] | 2.070 ± 0.110[b] | 0.963 |
| (b) Exponential model | | | |
| 30 | 13.89 ± 1.20[a] | 0.190 ± 0.007[a,b] | 0.986 |
| 40 | 10.56 ± 1.13[b] | 0.200 ± 0.009[a] | 0.991 |
| 50 | 11.71 ± 0.67[a,b] | 0.180 ± 0.003[b] | 0.994 |
| 60 | 11.42 ± 1.78[b] | 0.180 ± 0.009[b] | 0.969 |

Superscripts with different letters in the same column indicate significant differences ($P \leq 0.05$).

with stirring, pumping, pipe flow, spraying, and other processing operations, even mastication.[22,29] The parameters of eqs. (2) and (3) for the values of apparent viscosity at 100 $s^{-1}$ were presented in Table III. The variation of $\eta_{100}$ values with concentration could be well described by power law model and exponential model, because higher determination coefficients ($R^2$) ranged from 0.963 to 0.987 and 0.969 to 0.994, respectively. For the power model and exponential model, there was no specific trend for the parameter "$a$ ($a_1$, $a_2$)", "$b$ ($b_1$, $b_2$)," suggesting that between the concentration and the temperature might have a strong interaction[19]; in the exponential model, parameters $a_2$ and $b_2$ decreased at high temperatures (50–60°C), indicating a lower dependency of apparent viscosity on concentration. Apparent viscosity (100 $s^{-1}$) of soy protein spray-coating dispersions as a function of concentration is shown in Figure 2. In general, an increase in soy protein concentration increased the viscosity. This is due to the higher solid contents resulting from mainly molecular movements and interfacial film formation.[30] Similar observations have been reported by various workers.[20,28,31]

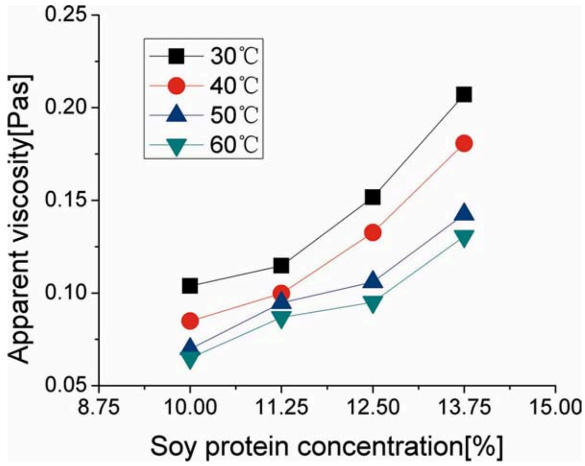


**Figure 2** Apparent viscosity (100 $s^{-1}$) of soy protein spray-coating dispersion at different concentrations and temperatures. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

### Influence of temperature

The temperature dependency of apparent viscosity could be described by the Arrhenius model,[19] and a shear rate of 100 $s^{-1}$ was used to calculate the apparent viscosity [from eqs. (2) and (3)]. The parameters are summarized in Table IV. The activation energy ($E_a$) at a constant share rate of the soy protein spray-coating dispersions was determined from the slopes of the lines in Figure 3. It is well known that the value of the activation energy reflects the sensitivity of the blend viscosity to temperature, and so the greater the activation energy, the more sensitive the behavior of the dispersions to temperature. As it is seen, $E_a$ decreased from 12.76 to 7.36 kJ/mol as the soy protein concentration increased from 10 to 11.25%, lower $E_a$ value of soy protein dispersions means that its apparent viscosity is less temperature-dependent; the values of $E_a$, which did not vary significantly at higher protein concentrations (12.5 and 13.75%), was 13.51 and 13.49 kJ/mol, respectively, indicating that there was no appreciable effect of concentration on $E_a$. A possible explanation for this phenomenon is that an increase in temperature can improve the capability of polymer segments to move, and the resistance between the melt layers decreases, thus causing a decrease in $E_a$ for the dispersions; while reached higher concentration (12.5 and 13.75%), molecular rearrangement is feasible, junction zones are more readily formed[26] and resulted in stiffer molecular chain, causing a higher $E_a$. Sharoba et al.[32] reported that the homogenized concentrate would have a higher $E_a$ than the nonhomogenized blend system, attributing to an increase in the number of insoluble particles, a decrease in particle size, and a decrease in viscosity. Collins and Metzger[33] observed that as the molecular weight of

TABLE IV
**Arrhenius Model ($\eta = A \exp(E_a/RT)$) for Temperature Dependency of Apparent Viscosity (at 100 $s^{-1}$) for Soy Protein Spray-Coating Dispersion at Different Concentrations**

| Concentrations (%) | Temperature dependency of parameters | | |
|---|---|---|---|
| | $A$ (Pa s) | $E_a$ (kJ/mol) | $R^2$ |
| 10 | $6.0329 \times 10^{-4}$ | 12.76 | 0.968 |
| 11.25 | $6.1114 \times 10^{-3}$ | 7.36 | 0.963 |
| 12.5 | $6.9202 \times 10^{-4}$ | 13.51 | 0.982 |
| 13.75 | $9.3796 \times 10^{-4}$ | 13.49 | 0.972 |

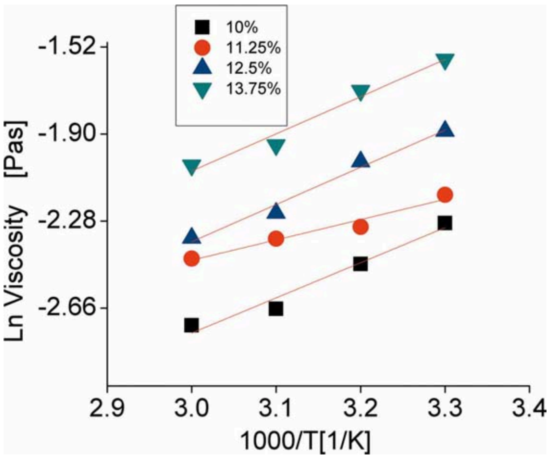


**Figure 3** Arrhenius plots for temperature dependency of apparent viscosities (100 $s^{-1}$) of soy protein dispersion. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

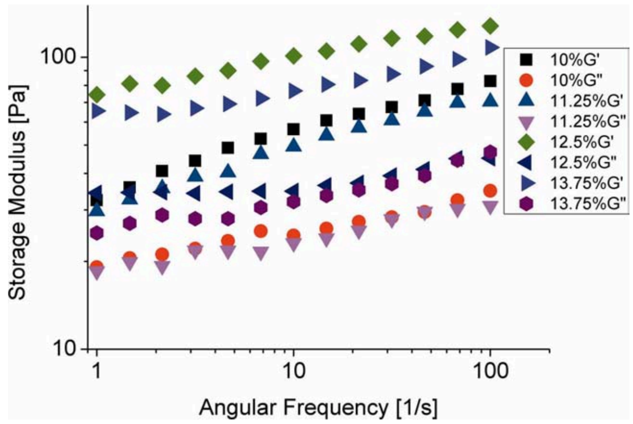


**Figure 4** Changes in storage ($G'$) and loss ($G''$) modulus during frequency sweep at 0.01% strain (50°C).[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the polymer increases, the influence of the temperature on the $E_a$ decreases. Obviously, apparent viscosity of spray-coating dispersions decreased along with the temperature increased (shown in Fig. 2). Increasing temperature, which caused increment of the thermal energy and the distance between molecules, destroyed the original structure of molecules and reduced the viscosity of the solution, leading to the decrement of the blend viscosity.[34–36]

## Dynamic shear rheological properties

The changes in storage modulus ($G'$) and loss modulus ($G''$) as a function of frequency of soy protein spray-coating dispersions with different concentrations at 50°C are shown in Figure 4. All the samples exhibited higher $G'$ than $G''$ values along the whole frequency range studied. The fact that both modules $G'$ and $G''$ increased with the rise of frequency suggested viscoelastic structure of the dispersions. The magnitudes of $G''$ did not overcome $G'$, that is, no cross-point of $G'$ and $G''$, indicating that the spray-coating dispersions exhibited a predominantly gel-like behavior ($G' > G''$).[37] These results suggested that the soy protein spray-coating dispersions were more elastic than viscous like.

Correlations between the magnitude of dynamic shear viscosity (complex viscosity, η*) and steady shear viscosity (apparent viscosity, η) at equal values of angular frequency (ω) and shear rate ($\dot{\gamma}$) were predicted by using the Cox–Merz rule.[22] This rule has been widely applied for studying polymers, complex food system, and film-forming dispersions.[7,38,39] Cox–Merz superposition of steady shear viscosity $\eta(\dot{\gamma})$, from large deformation measurements, and dynamic viscosity $\eta^*(\omega)$, from small deformation analyses, is consistent with topological entanglement interactions of individual species.[40] Figure 5 shows the apparent viscosity (η) and complex viscosity (η*) versus shear rate ($\dot{\gamma}$) and angular frequency (ω) logarithmic curves. Obviously, the values of complex viscosity (η*) were higher than apparent viscosity (η), demonstrating that the Cox–Merz rule was not applicable. Complex viscosity (η*) is almost 10 times higher than the apparent viscosity (η). The departures of Cox–Merz rule have been attributed to structure decay because of the affection of strain deformation[41] and also interpreted in terms of the interactions between polymeric chains, occurring in addition to entanglements and reflecting gel-like properties.[42] It could be concluded that the deviation from the Cox–Merz rule was due to the addition of additives (glycerol, CMC, and polycarboxylic salt).

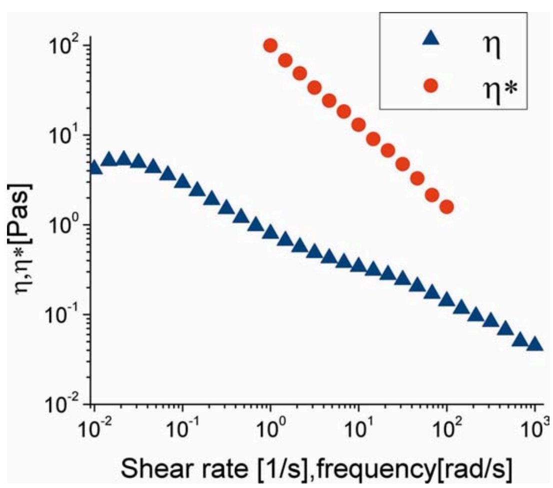


**Figure 5** Test of Cox–Merz rule for soy protein (13.75%) spray-coating dispersions (50°C).[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

**TABLE V**
**Mechanical Properties of Composite Paper**

| Samples | Thickness (mm) | Tensile strength (MPa) | % Elongation |
|---|---|---|---|
| S1 | $0.1557 \pm 0.0055^{b,c}$ | $16.45 \pm 1.15^{a}$ | $1.6 \pm 0.0^{a}$ |
| S2 | $0.1481 \pm 0.0061^{c}$ | $13.40 \pm 1.68^{b}$ | $1.6 \pm 0.0^{a}$ |
| S3 | $0.1564 \pm 0.0160^{b,c}$ | $17.24 \pm 1.84^{a}$ | $1.6 \pm 0.0^{a}$ |
| S4 | $0.1831 \pm 0.0110^{a}$ | $14.18 \pm 1.74^{b}$ | $1.6 \pm 0.0^{a}$ |

Superscripts with different letters in the same column indicate significant differences ($P \leq 0.05$).

Leveling behavior was determined by the linear viscoelastic properties of spray-coating dispersion. In general, high magnitudes of $G'$ resulted in surface leveling.[39] In our case, the spray-coating dispersion was more predominantly elastic properties than viscous behaviors, which could interpret the departure of the Cox–Merz superposition too. Thus, elasticity plays the main role in controlling soy protein-based film properties.

### Composite paper mechanical properties

Mechanical properties are of great importance for edible packaging materials; not only adequate strength ensures the integrity of packaging stuffs but reduces the appearance of defects such as pinholes.[7] Four groups of soy protein–celery composite paper S1 (10%), S2 (11.25%), S3 (12.5%), and S4 (13.75%) were prepared, according to the formula given in Table I, by spray-coating soy protein on celery paper. The celery paper sheet made with same technological parameters in a same day is called a same batch product. Because the performance between the different batches of celery paper are different, Five adjacent sections of celery paper in same batch are selected ,where one section is taken as control group and the other four sections are sprayed with four concentration levels of soy protein dispersions (Table I) separately. Then four kinds of soybean protein–celery composite paper are made, marked by S1′, S2′, S3′, S4′, respectively, in order to reduce the error. The mechanical properties′ values of composite paper are given in Table V. The thickness of composite paper varied from 0.1481 to 0.1831 mm. S4 exhibited higher thickness than others. TS accurately reflects the resistance of a material to a force tending to pull it apart, which indicates the ultimate force a material can sustain. The TS of soy protein–celery paper varied from 13.40 to 17.24 MPa, which is comparable with that of LDPE films (9–17 MPa); these values were substantially higher than those of soy protein films (3–9 MPa).[43] However, composite paper presented lower elongation rate than those of edible films. Figure 6 shows the comparison of thickness and TS between soy protein–celery composite paper and celery paper control. Pure celery paper control was thicker than coated paper, and the TS of celery paper was slightly higher than that of protein-coated paper, except for S4. These changes may result from the interactions between the biopolymers (celery fiber and soy protein), the solvent, plasticizers, and cross linking agent dispersed in the space of matrix.[44] The differentiations can also be attributed to different molecular structures.

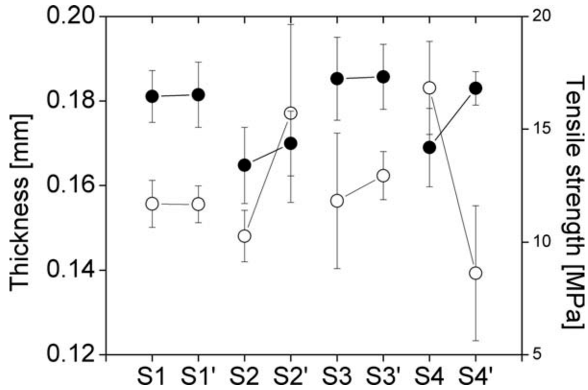


**Figure 6** Comparison between soy protein–celery composite paper and pure celery paper. Open symbol: thickness; closed symbol: tensile strength.

### Heat seal properties

The heat-sealing ability of the soy protein–celery composite paper was determined, and the optimum seal strength was achieved at 110–130°C during 5–7 s for all the samples. Figure 7 shows the heat-sealing strength of soy protein-coated paper (S1, S2, S3, and S4). It was observed that the cohesion of soy protein and celery fiber resulted in the sealing of celery paper, and the magnitudes of coated paper increased from 114.850 /m to 160.560 N/m with the increase of soy protein concentration from 10 to 13.75%. Pure celery paper control had no desired sealability due

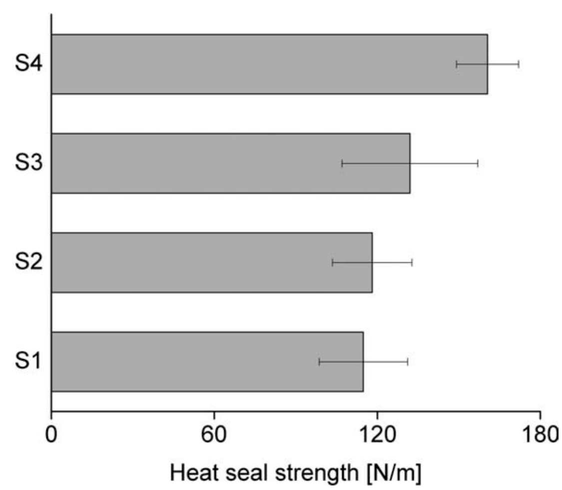


**Figure 7** Heat seal strength of soy protein-coated paper.

to their inherent structures such as the stuck celery fiber. The film-forming dispersion of soy protein is a kind of viscoelastic material that possess the dual characteristics of solid and liquid. These features are in close accordance with the rheological results presented earlier (steady-shear and dynamic-shear properties). Proteins are of sophisticated configuration because of their inter and intramolecular interactions and a lot of the side-chain blocks.[7] It is the visco feature that improves the heat-sealing properties of coated celery paper.

## CONCLUSIONS

Rheological studies showed that the soy protein spray-coating dispersions with different soy protein concentrations (10–13.75%) exhibited a shear-thinning behavior. The Herschel–Bulkley's model was adequate for describing the flow behavior of the soy protein dispersion at higher temperature (50 and 60°C). Dependence of concentration and temperature was clearly described by the power law model, exponential model, and the Arrhenius model. In terms of the frequency sweep data, the soy protein spray-coating dispersions were more elastic than viscous like. Because of the addition of additives (glycerol, CMC, polycarboxylic salt), the Cox–Merz rule was not applicable.

The cohesion of soy protein and celery fiber resulted in sealing of celery paper. The seal strength increased with the increase in soy protein concentration. The seal strength increased from 114.850 to 160.560 N/m, when the soy protein concentration increased from 10 to 13.75%.

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