

January 2015

## Evaluating an Ultrasonic Flaw Detector-Based Method to Characterize Solubility of Whey Protein Concentrate

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### Recommended Citation

Hauser, M. and Amamcharla, J. (2015) "Evaluating an Ultrasonic Flaw Detector-Based Method to Characterize Solubility of Whey Protein Concentrate," *Kansas Agricultural Experiment Station Research Reports*: Vol. 1: Iss. 8. <https://doi.org/10.4148/2378-5977.1155>

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# Evaluating an Ultrasonic Flaw Detector-Based Method to Characterize Solubility of Whey Protein Concentrate

## Abstract

One of the important characteristics of powders is their ability to quickly break down and disperse. Currently, the dairy foods industry does not have a reproducible method that can quantify this behavior. A previously developed method for an ultrasonic flaw detector was used to see if an ultrasound could characterize the dissolution of whey protein concentrate with a protein content of 80% (WPC80). For this study, an ultrasonic flaw detector equipped with a 1 MHz immersion transducer was operated in a pulse-echo mode. WPC80 was aged at 25°C and 40°C for 3 weeks. After powder addition, ultrasound, focus beam reflectance measurement (FBRM), and solubility index were taken at regular intervals for 30 minutes. The time of flight and amplitude of the first and second peak were collected so that the velocity and attenuation could be calculated. Graphs of velocity and attenuation over time showed that there were differences between powders stored at different temperatures. Powders stored at lower temperatures had higher ultrasound velocities at 1800 seconds. From the attenuation data, the peak height, peak time, and area under the attenuation were extracted. The FBRM and solubility index showed that powders stored at 25°C were more soluble and these powders had a higher relative velocity at 1800 s, smaller area under the attenuation curve, lower peak height, and higher peak time. Overall, an ultrasonic flaw detector can be used for examining dissolution behavior of WPC80.

## Keywords

ultrasonic flaw detector, whey protein concentrate, solubility

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## **Evaluating an Ultrasonic Flaw Detector-Based Method to Characterize Solubility of Whey Protein Concentrate**

*M. Hauser and J. Amamcharla*

### **Summary**

One of the important characteristics of powders is their ability to quickly break down and disperse. Currently, the dairy foods industry does not have a reproducible method that can quantify this behavior. A previously developed method for an ultrasonic flaw detector was used to see if an ultrasound could characterize the dissolution of whey protein concentrate with a protein content of 80% (WPC80). For this study, an ultrasonic flaw detector equipped with a 1 MHz immersion transducer was operated in a pulse-echo mode. WPC80 was aged at 25°C and 40°C for 3 weeks. After powder addition, ultrasound, focus beam reflectance measurement (FBRM), and solubility index were taken at regular intervals for 30 minutes. The time of flight and amplitude of the first and second peak were collected so that the velocity and attenuation could be calculated. Graphs of velocity and attenuation over time showed that there were differences between powders stored at different temperatures. Powders stored at lower temperatures had higher ultrasound velocities at 1800 seconds. From the attenuation data, the peak height, peak time, and area under the attenuation were extracted. The FBRM and solubility index showed that powders stored at 25°C were more soluble and these powders had a higher relative velocity at 1800 s, smaller area under the attenuation curve, lower peak height, and higher peak time. Overall, an ultrasonic flaw detector can be used for examining dissolution behavior of WPC80.

Key words: ultrasonic flaw detector, whey protein concentrate, solubility

### **Introduction**

Whey protein concentrate is added to a variety of food products, such as high protein bars and beverages, to improve the nutritional and functional properties of the finished food product. When choosing a WPC, a manufacturer considers the solubility of the powder. If the powder is not soluble, then pipes and filters can become clogged, a sediment layer forms, and the finished product will not have the desired nutritional or functional properties. A variety of tests can be used to determine the solubility, but these tests are subjective, time-consuming, and difficult to reproduce. Literature has shown that ultrasound spectroscopy is a rapid and precise method to characterize the solubility of dairy-based powders; however, ultrasound spectroscopy requires expensive equipment and skilled technicians. An economical alternative is

to use an ultrasonic flaw detector (UFD), which is commonly used in the construction industry to detect flaws and defects in metals and structures. For this study, the objective was to determine if a UFD-based method could characterize the solubility of WPC.

## Experimental Procedures

Two batches of WPC80 were obtained from a commercial manufacturer and stored at 25°C and 40°C for 3 weeks. The powders were evaluated on day 0, day 3, week 1, week 2, and week 3. In a 1 liter beaker, 19.76 grams of WPC were added to 375 grams of water with an overhead stirrer at 900 rotations per minute. After all the powder was added, the overhead stirrer was set at 400 rotations per minute, and ultrasound and FBRM data were collected every 15 seconds and 10 seconds, respectively.

A 1 MHz immersion transducer was attached to a UFD, which operated in a pulse-echo mode. A custom-built stainless steel holder kept the transducer a constant distance from the reflector plate. Figure 1 shows the experimental setup (A) and the immersion transducer holder (B). The experimental setup consisted of UFD (Epoch LTC, Olympus Scientific Solutions, Waltham, MA); focused beam reflectance measurement (FBRM) (Particle Track E25, Mettler Toledo, Columbus, OH); an immersion transducer (V303-SU; Olympus Scientific Solutions, Waltham, MA) in a holder; and a four bladed overhead stirrer (Caframo, Georgian Bluffs, Ontario, Canada). The powder dissolution temperature was maintained at 40±0.1°C with a temperature-controlled water bath (Fisher Scientific, Pittsburgh, PA).

From the time-of-flight and amplitude collected with the UFD, the relative ultrasound velocity and the attenuation of the ultrasound wave were calculated. To quantify the solubility of WPC, the relative ultrasound velocity at 1800 seconds was extracted from the relative ultrasound velocity data; and the area under the attenuation curve, peak height, and peak time were extracted from the attenuation data. Focused beam reflectance measurement and solubility index were used as reference methods. The FBRM categorized the particles based on their size into fine (<10 micrometers), medium (10-50 micrometers), and large (50-150 micrometers) particles. The maximum count for fine and medium particles, minimum count for large particles, as well as the time to reach the maximum and minimum count were examined to characterize the solubility of WPC. The solubility index was determined by centrifuging 15 milliliters of sample at 700 × gravity for 10 minutes and calculating the total solids for the supernatant.

## Results and Discussion

After powder addition, the ultrasound signal disappeared due to water entering the powder particles and subsequently releasing air into the solution. Over time, the ultrasound signal reappeared and the relative ultrasound velocity stabilized. Figure 2 shows the relative ultrasound velocity for day 0 powder and powders that were stored at 25°C and 40°C for 3 weeks. The powders stored at 25°C for 3 weeks had a higher relative ultrasound velocity than the day 0 powder and the powder stored at 40°C for 3 weeks. The relative ultrasound velocity decreased after 3 weeks of storage at 40°C.

Figure 3 shows how the relative velocity at 1800 seconds changed over the storage period for both storage temperatures. The graph shows that powders stored at 25°C had a higher relative ultrasound velocity at 1800 seconds than powders stored at 40°C. As the storage time increased, the relative ultrasound velocity at 1800 seconds increased for powders stored at 25°C and decreased for powders stored at 40°C. A significant difference between the two storage temperatures was noticed on weeks 2 and 3.

In addition to velocity, the attenuation was examined. Figure 4 shows the attenuation trend for day 0 powder and powders stored at 25°C and 40°C for 3 weeks. The day 0 powder and powder stored at 25°C for 3 weeks had a trend of increasing, reaching a peak and then decreasing. The powder stored at 40°C for 3 weeks had a trend of increasing, reaching a peak, decreasing, and then gradually increasing again. The differences in the trend led to changes in the area under the attenuation curve, peak height, and peak time. Figure 4 shows the changes in area under the attenuation curve (A), peak height (B), and peak time (C) over the storage period for both of the storage temperatures.

After day 0, the area under the attenuation curve increased from 49.84 to 90.49  $\text{Np} \times \text{seconds/millimeter}$  for powders stored at 40°C for 3 weeks and decreased from 49.84 to 47.01  $\text{Np} \times \text{seconds/millimeter}$  for powders stored at 25°C for 3 weeks. A significant difference in the area under the attenuation curve was observed between the two storage temperatures on week 2 and week 3. On all the experimental days, the powders stored at 40°C had a higher peak height than powders stored at 25°C. Week 2 was the only time point that had a significant difference between the two storage temperatures. With the peak time, little change was observed from day 0 to week 3. Powders stored at 25°C typically had a higher peak time than powders stored at 40°C, and a significant difference between the two storage temperatures was noticed on week 3.

With the FBRM, the large particles had a trend of decreasing as the particles disintegrated in fine and medium particles; which subsequently increased the count for fine and medium particles. Figure 6 shows how the fine (A), medium (B), and large (C) particle count changed over 1800 seconds for day 0 powder and after 3 weeks of storage at 25°C and 40°C. The large particle count at 1800 seconds for day 0 and powders that had been stored at 25°C for 3 weeks were similar. However, the time to reach this count increased from 1000 seconds for day 0 to 1500 seconds for powders stored at 25°C for 3 weeks. Storing the powders at 40°C led to an increase in large particle count at 1800 seconds. The large particle count increased from 400 for day 0 powder to 900 for powders stored at 40°C for 3 weeks.

The medium particle count at 1800 seconds was similar for day 0 powder and powders that were stored at 25°C for 3 weeks. For day 0 powder and powders stored at 40°C for 3 weeks, the counts were 12,000 and 9,000, respectively. The fine particles had a general trend of increasing rapidly and then stabilizing or increasing at a slower rate. As the storage time and temperature increased, the fine particle count at 1800 seconds declined. On day 0, the fine particle count was 20,000 and after 3 weeks of storage the count decreased to 17,000 and 14,000 for powders stored at 25°C and

40°C, respectively. Overall, the increasing storage time and temperature led to a reduction in solubility.

Figure 7 shows how the solubility index at 1800 seconds changed for WPC on each experimental day at both storage temperatures. From day 0 to week 3, the powders stored at 25°C and 40°C had a 3.99% and 3.36% reduction in solubility, respectively. However, no significant differences were noticed between the two storage temperatures.

Out of all the ultrasound parameters, the relative velocity at 1800 seconds and the area under the attenuation curve were the most sensitive to changes between the two storage temperatures. The differences in the two parameters were compared to the FBRM data. The UFD was not compared to the solubility index data since the solubility index did not detect differences between the two storage temperatures. The FBRM data showed that the solubility decreased as the storage time and temperature increased. The relative velocity at 1800 seconds was higher for more soluble powders, and the area under the attenuation curve increased for less soluble powder. Therefore, a soluble WPC had a larger relative velocity at 1800 seconds and a lower area under the attenuation curve.

## Conclusions

The relative ultrasound velocity at 1800 seconds and area under the attenuation curve were able to detect differences in solubility starting at week 2; whereas the solubility index was not able to detect any differences between the solubility of the powders. The FBRM showed that the solubility decreased as the storage time and temperature increased. Therefore, a soluble WPC had a high relative ultrasound velocity at 1800 seconds and a low area under the attenuation curve. Overall, a UFD can characterize the solubility of WPC80.

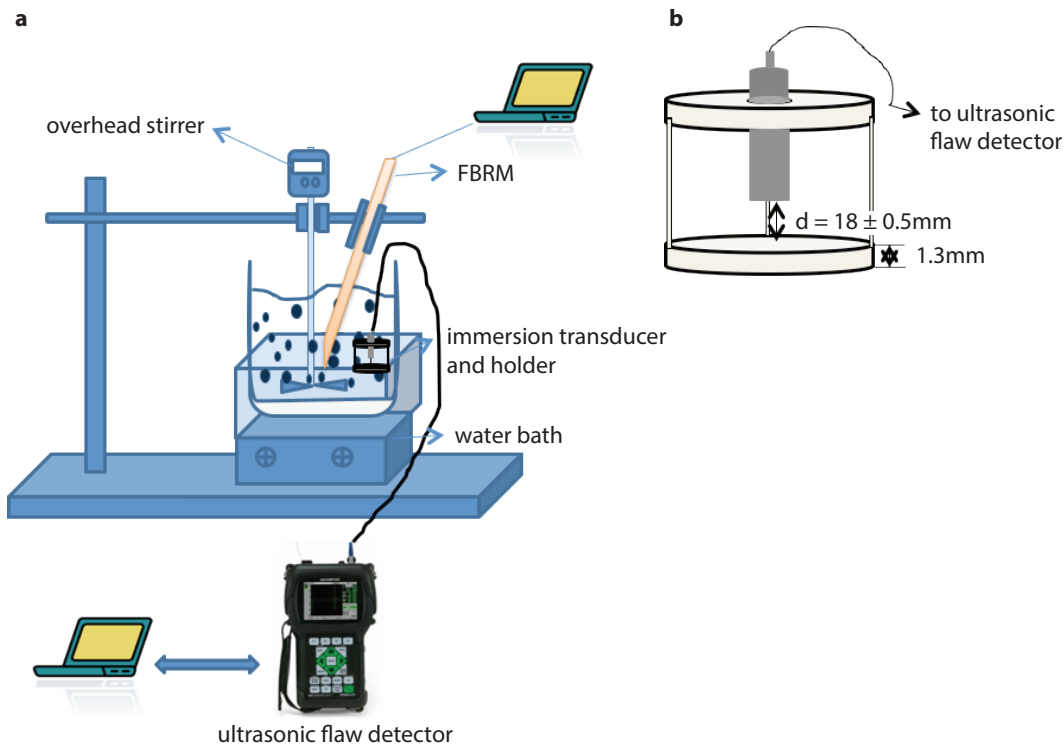


Figure 1. Experimental setup used for characterizing powder dissolution (a); Immersion transducer holder (b).

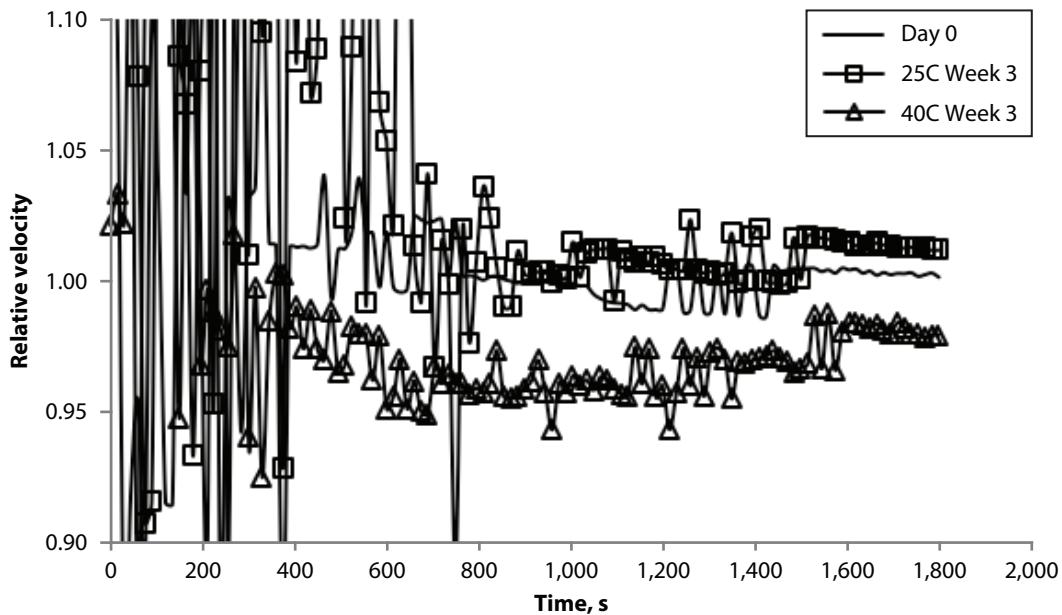


Figure 2. Relative velocity for WPC on day 0 and after 3 weeks of storage at 25°C and 40°C.

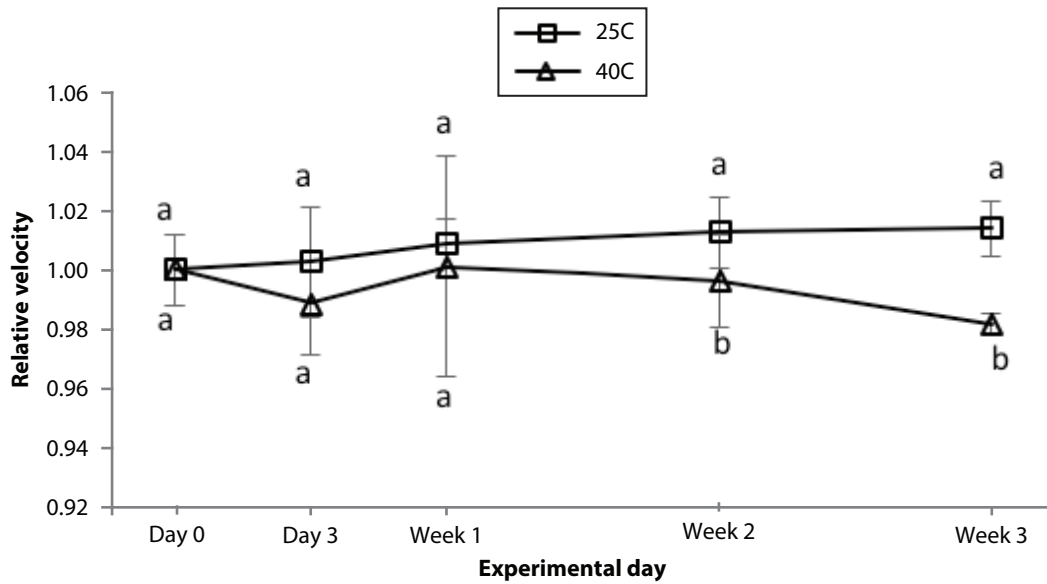


Figure 3. Relative velocity at 1800s for WPC on each experimental day for powders stored at 25°C and 40°C. The error bars represent the standard error, and data points with different letters differ between storage temperatures ( $P < 0.05$ ).

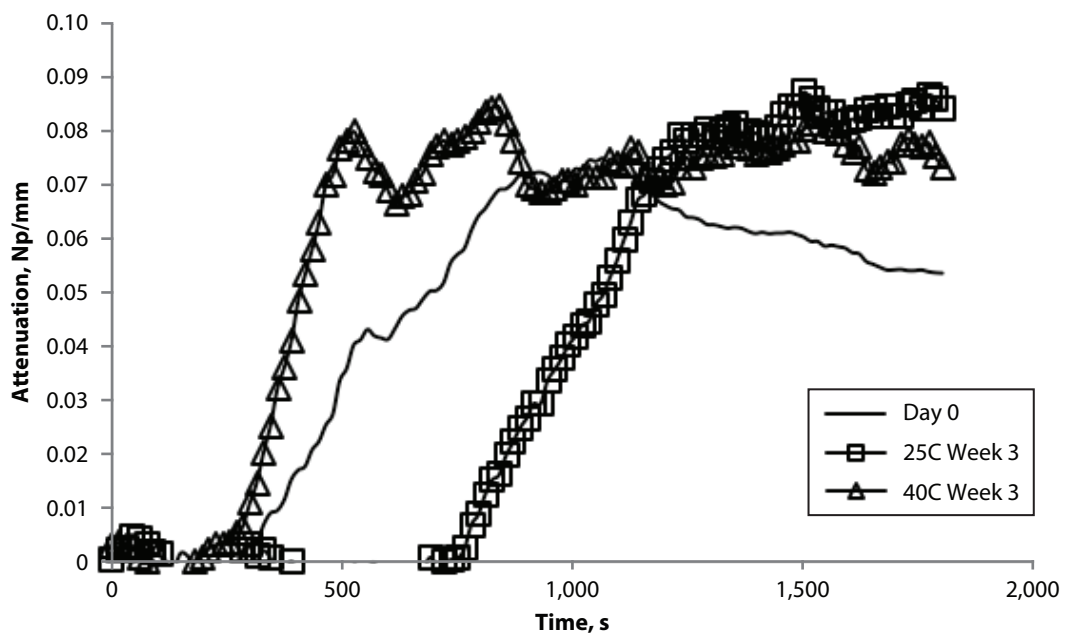


Figure 4. Ultrasound attenuation of WPC on day 0 and after 3 weeks of storage at 25°C and 40°C.



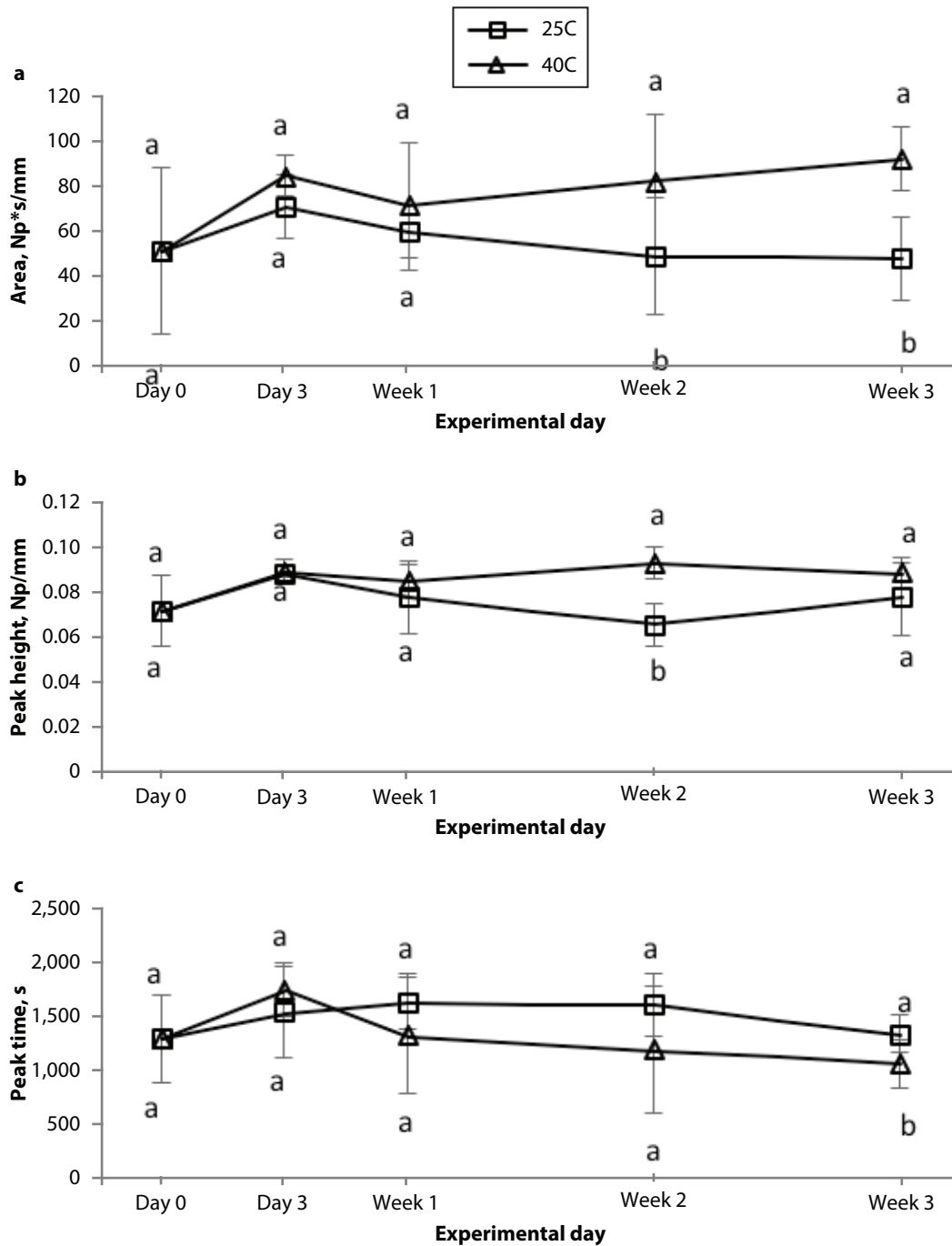
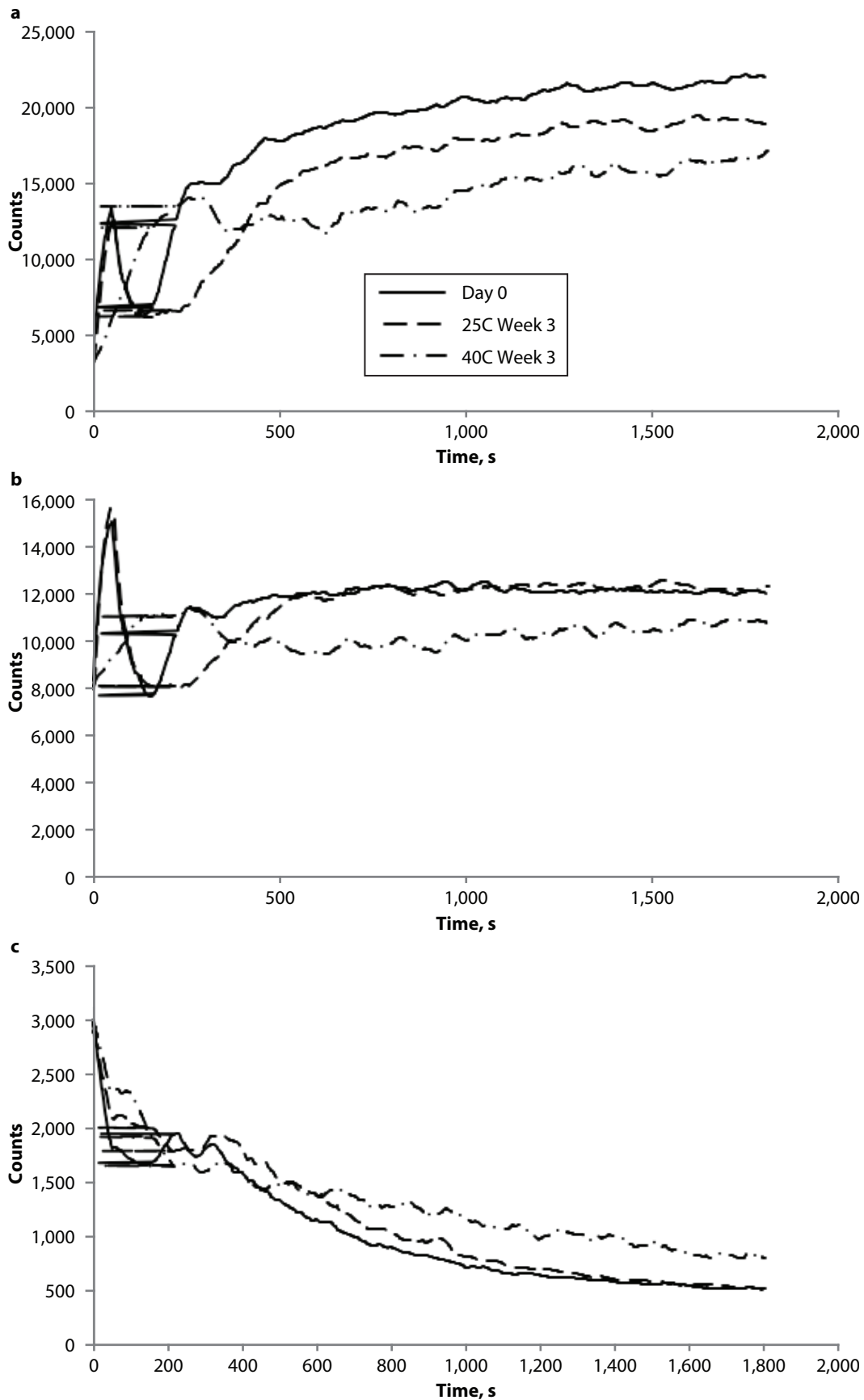


Figure 5. Area under the attenuation curve (A), peak height (B), and peak time (C) on each experimental day for WPC stored at 25°C and 40°C. The error bars represent the standard error, and data points with different letters differ between storage temperatures ( $P < 0.05$ ).



**Figure 6. Change in WPC particle counts during an experiment for fine (A), medium (B), and large particles (C) for day 0 powders and powders that have been stored at 25°C and 40°C for 3 weeks.**

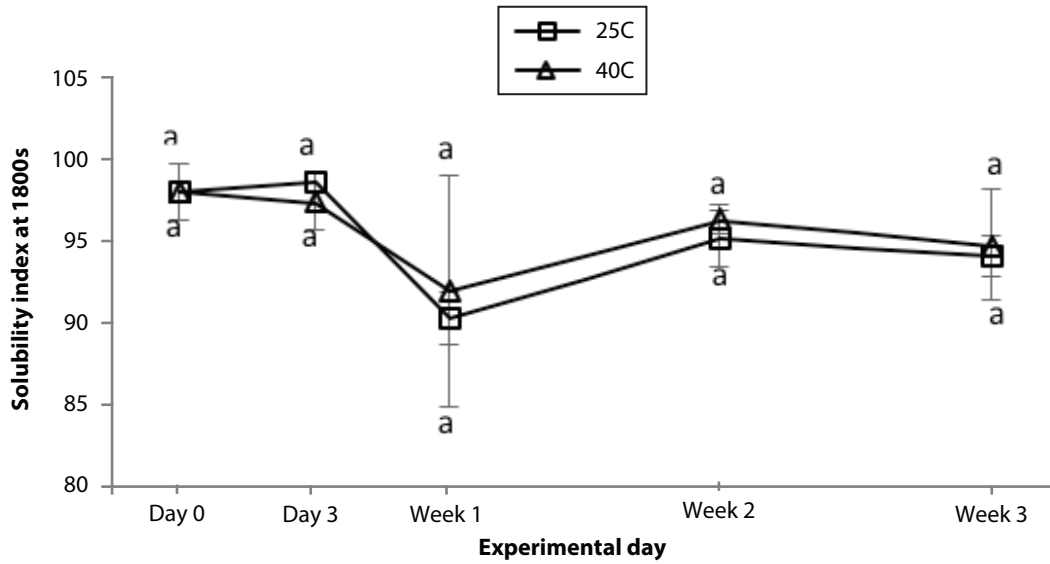


Figure 7. Solubility index at 1800 seconds on each experimental day for WPC stored at 25°C and 40°C. The error bars represent the standard error, and data points with different letters differ between storage temperatures ( $P < 0.05$ ).