Data Variability in Rheological Measurement of Semi-Solid Foods: Effects of Loading Normal Force

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Received: 11.3.2002, Final version: 2.11.2002

Abstract:
Previous studies involving rheological measurement of semi-solid foods have reported a large amount of data variability, but have focused little on understanding the cause of such variability. This project examined whether differences in normal force have an effect on the variability of rheological measurements. Experimental methods focused on error introduced during sample loading; specifically whether normal force application during loading influenced the storage (G') and loss (G") moduli of semi-solid and liquid foods. Samples were loaded to 5 or 20 N between the parallel plates of a TA-1000N rheometer and tested immediately. For all semi-solid products tested, normal force application during sample loading did significantly affect oscillatory parameters, with G' and G" measurements increasing up to 50 % with greater normal force. However, loading normal force did not significantly influence the parameters measured for the liquid sample. This suggests that differences in normal force during loading could be a significant source of data variability during rheological measurement of semi-solid products.

Zusammenfassung:
Die meisten Studien, bei denen rheologische Untersuchungen an halbfesten Lebensmitteln durchgeführt wurden, berichten von stark streuenden Daten, haben aber wenig zum Verständnis dieses Phänomens beigetragen. In dieser Arbeit wird untersucht, ob unterschiedliche Normalkraftbeladungen einen Effekt auf die Streuung der rheologischen Messungen haben. Die experimentelle Arbeit fokussiert auf den Fehler, der während der Einfüllphase auftreten kann; insbesondere ob das Aufprägen von Normalkräften in dieser Phase die Speicher- und Verlustmodule von halbfesten und flüssigen Lebensmitteln beeinflusst. Zwischen parallelen Platten eines TA-1000N Rheometers wurden Proben mit 5 bis 10 N belastet und sofort getestet. Für alle getesteten halbfesten Proben werden die oszillatorischen Parameter durch das Aufprägen von Normalkräften beeinflusst, wobei G' und G''-Werte mit wachsender Normalkraft um bis zu 50% ansteigen. Für flüssige Proben gilt dies jedoch nicht. Das legt die Vermutung nahe, dass die beobachtete Variation in den gemessenen Daten für halbfeste Produkte durch angreifende Normalkräfte während der Einfüllphase verursacht werden.

Résumé:
Les précédentes études concernant les mesures rhéologiques de produits alimentaires semi solides ont montré une grande part de dispersions dans les données, mais ne se sont pas souciées de comprendre les causes d'une telle dispersion. Ce projet a recherché à savoir si les différences de force normale avaient un effet sur la dispersion des mesures rhéologiques. Les méthodes expérimentales se sont focalisées sur l'erreur introduite par l'insertion de l'échantillon; spécifiquement, est-ce que l'application de forces normales durant la mise en place de l'échantillon influence les modules élastiques (G') et de perte (G'') de produits alimentaires semi solides et liquides. Les échantillons furent insérés en utilisant des forces de 5 à 20 N entre les plans parallèles d'un rhéomètre TA-1000N et testés immédiatement. Pour tous les produits semi solides testés, l'application de forces normales durant l'insertion a affecté de manière significative les paramètres oscillatoires, avec des mesures de G' et G'' augmentant jusqu'à 50% pour les forces normales les plus grandes. En revanche, la force normale n'a pas influencé de manière significative les paramètres mesurés pour l'échantillon liquide. Ceci suggère que les différences de force normale pendant l'insertion peuvent être une source significative de dispersion de données durant la mesure rhéologique de produits semi solides.

Key words: rheology, normal force, loading, semi-solid, food

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ent at all 3 frequencies. For example, at 1 Hz, G' values for bologna were 17,923 and 23,675 Pa when loaded to 5 and 20 N, respectively. For every semi-solid sample, the G' values were higher when loaded to 20 N compared to loading at 5 N, even though the differences were not statistically significant for the cheddar cheese at 10 Hz or for either cheese at 1 Hz.

Looking at the ratio of G' for each of the products, the trend is very consistent among different frequencies for each product. For bologna, the ratio ranges from 1.25 to 1.32, indicating fairly consistently that G' can change about 30 % due to differences in loading normal force. Similarly, the ratio for mozzarella cheese is somewhat less, ranging from 1.11 to 1.13, but is still very consistent, suggesting that G' may change about 12 % due to differences in loading normal force. The ratio for cheddar cheese ranged from 1.00 at the lowest frequency to 1.08 at the highest frequency, suggesting that the effect of normal force on cheddar cheese is not as great as the other two semi-solid products.

Table 4 shows the G" values for the three semi-solid products. The effect of normal force on G" values was very similar to the effect on G'. In every case, G" for samples loaded to 20 N normal force was greater than for samples loaded to 5 N. Statistically, the bologna samples were significantly different at all frequencies, the mozzarella only at 100 Hz, and the cheddar samples were not statistically different due to loading normal force. The trend in the ratios was very consistent, however, and was similar to the ratios seen with G'. For bologna, the ratios of G" (20N/5N) ranged from 1.27 to 1.33, suggesting again that loading normal force can change G" values by 30%. For mozzarella cheese, the G" ratios ranged from 1.14 to 1.16, indicating that normal force affected G" values by about 15%. And in the cheddar cheese, ratios ranged from 1.05 to 1.08, suggesting that there is a consistent effect on G" from loading normal force, even though statistically the differences were not significant.

3.3 EFFECT ON MUSTARD

Since mustard is a liquid product, it was not possible to apply the same forces as were applied to the semi-solid products, hence mustard samples were subjected to normal forces of 1 or 5 N. In general, mustard samples showed slightly greater G' values for samples loaded to greater normal force, but this trend was much less dramatic for the mustard samples than the semi-solid samples. Statistical analysis verified that there was not a significant difference in G' value based on different loading normal forces ($p = 0.9640$). The liquid product, mustard, responded to normal force quite differently than the semi-solid products, and there were only minimal differences in compactness detected between samples loaded to 1 or 5 N.

4 CONCLUSIONS

This study confirmed that loading normal force does influence the oscillatory measurements of semi-solid food products. Specifically, higher normal force (20 N) applied during sample loading produced G' and G" values 5 - 50 % higher than samples loaded to the lower normal force of 5 N. Results suggest that if samples are not loaded to the same specified normal force for each test, data variability will be introduced during loading. Additional, on-going studies regarding the response of semi-solids to loading normal force (i.e. investigation of sample relaxation) suggest that effects of loading normal force on some semi-solid samples are not eliminated or minimized by simply allowing the sample to rest prior to testing. Thus, strict sampling protocols, which control and/or measure normal force during loading are recommended. This study also demonstrated the large differences in response to loading normal force between liquid and semi-solid products. This conclusion is significant for rheological testing because the methods adopted for improving repeatability in results for liquid products may prove ineffective when used for testing semi-solid products. Future research is needed to develop methods designed to minimize data variability during rheological testing of semi-solid products.

ACKNOWLEDGEMENT

This work was supported by the Oklahoma Agricultural Experiment Station.

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