

Spotlight on Physical Testing

By Larry Gades

Walk into a Physical Testing Lab for the first time and you will be confronted by an array of instruments with few, if any, which are recognizable. The testing application of many instruments could well remain a mystery even upon closer inspection.

The goal of this article is to unravel the mysteries of Physical Testing and provide an overview of how these techniques are used in the food industry.

Several different disciplines are utilized to define the physical properties of foods and ingredients. The use of these disciplines provides critical information about the functional properties and appearance of ingredients and foods. The sections below briefly describe these disciplines and provide examples on their use.

COLOR AND COLOR APPEARANCE

Color appearance is a critical consumer acceptance attribute. Controlling the color of incoming ingredients and finished products can be critical to the success of a product. Medallion Laboratories offer both Hunter

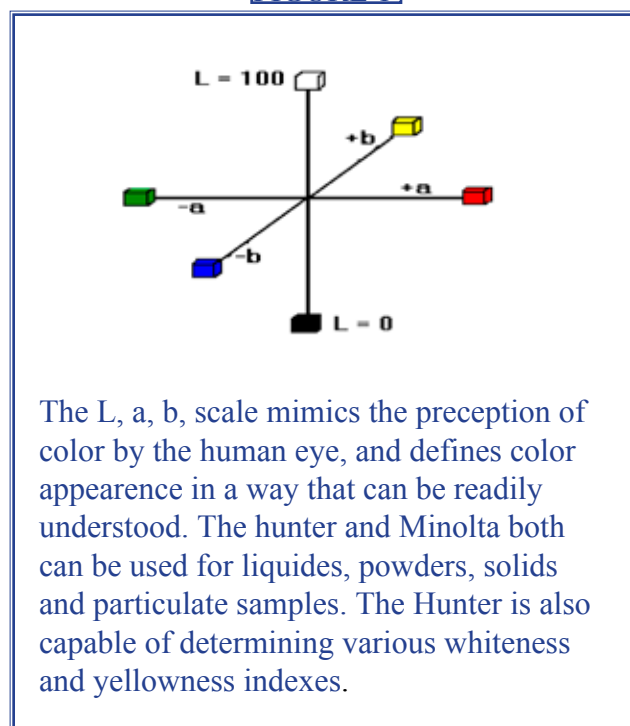
and Minolta color analysis instruments using the L,a,b color scale. The L,a,b color scale is depicted graphically in figure 1.

The Lovibond method is the standard color method for measuring the color of refined oils and melted shortenings. A visual match is made between the sample and color filters with varying indexes of red

and yellow color.

Other methods for measuring color are also available. Spectrophotometric methods are widely used. The ASTA has published several standard methods for spices and spice extracts. Methods for colorants are

FIGURE 1



available and should be referenced when submitting samples for analysis.

WHEAT AND FLOUR QUALITY TESTS

A number of specialty instruments and test procedures have been developed to measure various quality as-

pects of wheat and wheat flours.

Many were designed for hard wheat flours to characterize critical factors in the production of developed yeast raised doughs. It is not possible to list all of the methods that have been developed, but a few are briefly described below. These are standard procedures published by the AACC (American Association of Cereal Chemists) in a two volume set titled American Association of Cereal Chemists Approved Methods.

The Brabender Farinograph consists of a dough-mixing chamber attached to a torque recorder. A standard weight of flour is placed in the mixing chamber and water is titrated into the flour to produce dough of optimum consistency. The amount of water required to make dough of optimum consistency is defined as the Absorption and is calculated as the water weight divided by the flour weight, expressed as a weight percent. The mixing time required to reach optimum consistency (peak time) is a second primary measure obtained. A number of other parameters relating to dough quality can also be calculated from the graph.

The Brabender Amylograph is a recording viscometer that can be programmed to heat, hold a temperature, or cool a slurried sample. Flour, ground wheat or starch samples are mixed with a buffer solution, then heated in the viscometer. The viscosity rises rapidly and reaches a peak as the starch is gelatinized. The nature of the peak viscosity is an indicator of α -Amylase activity due to sprout damage in untreated samples, or it can be an indicator of malt addition in treated flours. A method is also available to test for levels of fungal enzymes. Other measurements that can be made are the gelatinization temperature, breakdown rate after gelatinization, and viscosity setback upon cooling.

The Falling Numbers apparatus also is an empirical measure of α -Amylase activity in ground grain or flour. This procedure takes less time to run than the Amylograph, but the extended data that can be obtained from the Amylograph cannot be obtained from the Falling Numbers apparatus. A seven-gram sample is mixed with 25 mL of water in a Falling Numbers tube. The slurry is mixed while being rapidly heated in a boiling water bath to gelatinize the starch. A timer then automatically records the time it takes for a probe to fall a fixed distance through the slurry. Higher levels of α -Amylase activity reduce the viscosity of the slurry and

also reduce the time for the probe to fall.

The Rapid Visco Analyzer (RVA) is a newer instrument that operates similarly to the Amylograph. A much smaller sample and slurry volume are used, so faster heating rates can be realized. Standard heat-hold-cool profiles for flours and starches require about 20 minutes to complete, compared to typical analysis times of 45 to 90 minutes for the Amylograph.

The Glutomatic gluten quality analyzer provides a quantitative measure of gluten. The instrument automatically washes the gluten from a dough ball, and the washed gluten is then centrifuged. The wet gluten is weighed and then dried. The wet and dry gluten percentages are reported. Also, a gluten index can be calculated from the ratio of wet gluten that passes through a screen in the centrifuge.

“Viscosity of Acidulated Flour-Water Suspensions” is a test that measures the relative gluten strength in soft wheat flours. This test was commonly called the MacMichael test in the past. But the MacMichael viscometer is no longer manufactured and the method has now been adapted to the Brookfield viscometer. Lactic acid is added to a suspension of flour in water and the viscosity is measured. The lactic acid causes the gluten proteins to coagulate, thickening the slurry giving a high viscosity reading. For many soft wheat flour applications, a strong gluten presence is a negative performance factor, and a low viscosity is desired.

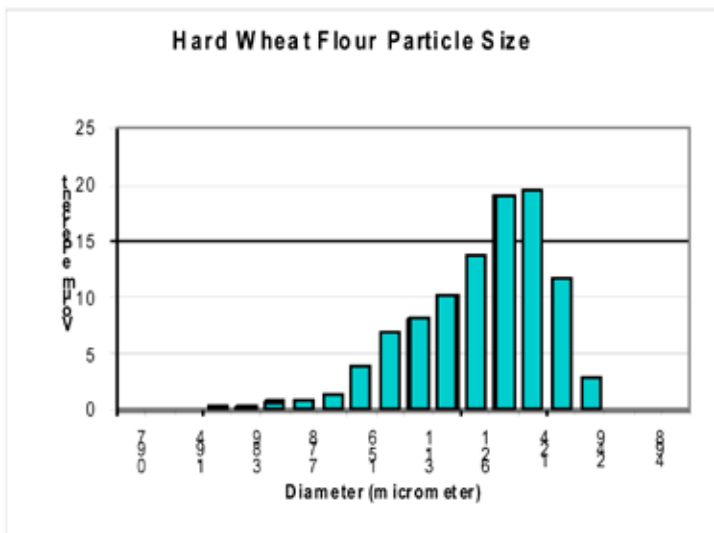
A recent addition to the AACC methods is a “Solvent Retention Capacity” method. This method measures the liquid holding capacity of flour in four different solvents. The different solvents were adopted to predict flour performance in different baking applications. Refer to the AACC method for a complete description.

PARTICLE SIZE ANALYSIS

Particle Size Analyses have traditionally been done using sieves with a range of mesh sizes. The Rotap Sieve Shaker is the standard for routine sieve sizing. A full range of sieves with meshes ranging from one inch to #400 (400 openings per square in, each 0.037 mm) are available.

Some powder samples tend to be sticky and blind (cling to or near) the openings of the sieves. When this happens, accurate results can not be obtained. The Alpine Air Jet Sieve Tester is used for these samples. The unique action of the Alpine prevents sieve blinding. However, only one sieve at a time can be used with the Alpine, which makes it a more time consuming test than the Rotap if a number of sieves are to be used.

A newer means of characterizing particle size is with a laser diffraction instrument. These instruments provide a complete size distribution without the physical separation required in sieve analysis. The Microtrac SRA 150 instrument defines particle size distributions in the range from 0.7 to 700 micrometers. The sub-micrometer lower limit allows much smaller particles



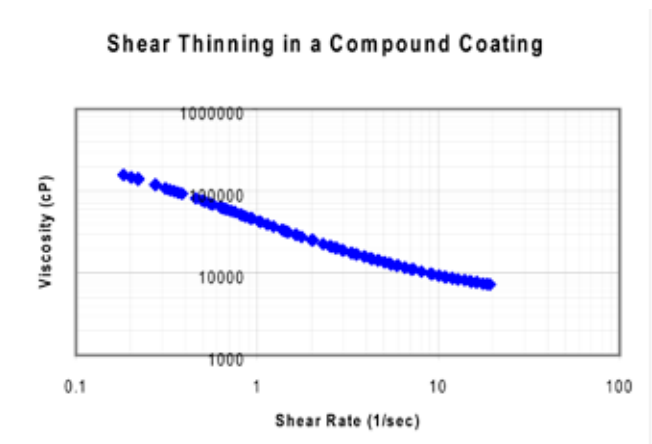
to be sized than can be done with sieve analysis. Particles are dispersed in a compatible liquid (a liquid that does not dissolve or agglomerate the particles). This allows analysis of emulsions and particles in suspensions, pastes and creams, as well as in powders.

OBJECTIVE TEXTURE TESTING

Many specialty instruments have been developed over the years to measure the texture of specific products. The Penetrometer used for pastes and soft solids and the Kramer shear press for peas and other vegetables are just two of a large number of instruments used in the food industry. In the 1960's scientists also began using the Instron Universal Testing Machine, which was and is widely used for materials testing, and developed methods to test a wide range of product textures.

A unique texture analyzer developed by Stable Micro-Systems is promoted as a universal texture testing machine, in that the software designed for a PC interface was developed specifically to automate most of the common texture analyses. A full range of test probes and fixtures have also been developed to allow testing a wide range of food products.

When analyzing texture it is generally necessary to test a number of pieces and determine the average to obtain a representative result. Techniques such as mixing or homogenizing the sample to obtain a representative



sample as is typically done for other methods cannot be done without altering the sample's texture. Therefore it is necessary to test samples as is and deal with the variability by making multiple measurements. Because of this, texture analysts will often ask for a surprisingly large amount of sample.

VISCOSITY

Many segments of the food industry have developed specialized instruments to analyze the viscosity and/or consistency of liquids and semisolids. The Bostwick Consistometer, widely used in the tomato industry and the Bloom Gelometer used to analyze gelatin gel strength are common examples. Many other parts of the food industry have developed procedures using general-purpose viscometers, such as the Brookfield. Equipped with standard straight spindles, T-bar spindles, low volume accessory, and low viscosity accessory the Brookfield becomes a versatile tool for many applications.

The specialty instruments listed above are empirical in-

struments, measuring an index of viscosity rather than measuring fundamental viscosity or rheological properties. For solving a problem with product viscosity, it is often necessary to use a more sophisticated viscometer to measure the complex flow properties often encountered with foods.

The modern controlled stress rheometer is a valuable tool for measuring fundamental rheological properties. These versatile instruments can operate in controlled stress, controlled rate and oscillation modes to compare and contrast these complex properties. When long chain food polymers, such as starches, gums and proteins, are present in foods they can impart complex flow properties. The most common flow properties consist of shear dependent effects (shear thinning and yield value are examples) and time dependent effects (thixotropy). Elasticity can also be an important aspect of a sample's rheology. Controlled stress rheometers can characterize all of these flow properties to help explain determine process control issues and product texture problems.

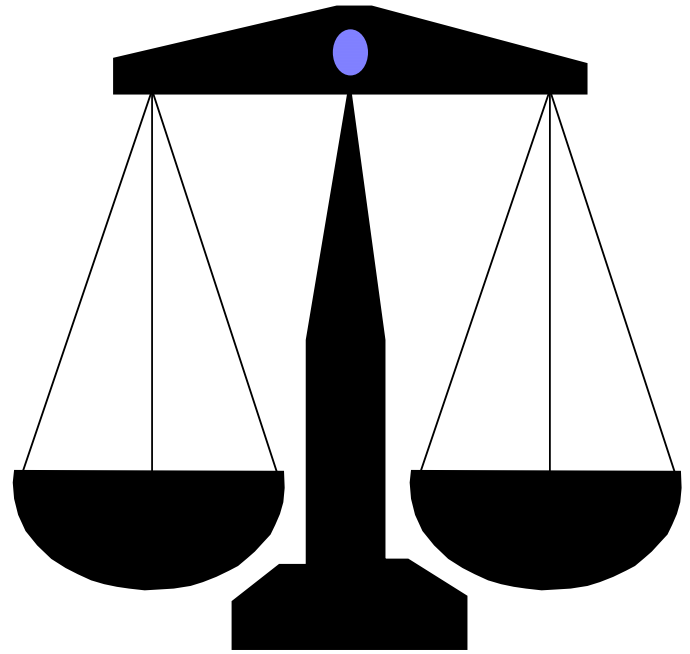
The Haake rheometer has a precise temperature control system that not only provides accurate temperature control, but allows samples to be heated or cooled at programmed rates. Viscosity as a function of temperature can be precisely measured.

THERMAL ANALYSIS

Thermal Analysis comprises a series of analytical techniques evaluating changes in samples as a function of temperature, or if done at a constant temperature, evaluating changes as a function of energy over time. These techniques include Differential Scanning Calorimetry (DSC) and Thermo-Gravimetric Analysis (TGA).

DSC has been the most widely applied thermal analytical technique to foods. A small portion of the sample is heated or cooled at a specified rate along with an empty reference pan. Any energy input difference required to keep the two pans at the same temperature is recorded as a change in heat flow. These changes are plotted versus temperature, and appear as endothermic peaks, exothermic peaks, or baseline transitions. Examples of endothermic peaks include crystals melting, starch gelatinizing, and proteins denaturing. Crystallization of a compound is an example that gives an exothermic peak. A baseline shift occurs when the sample under-

goes a change in heat capacity, for example, the temperature at which a plastic undergoes a glass transition from a hard glass to a soft glass state.



Starch gelatinization and glass transitions are probably the most studied DSC applications in Foods. Every starch type has a unique DSC fingerprint that includes the gelatinization onset temperature, peak temperature and total energy consumed. Glass transitions have been studied in a wide variety of food products. Denaturation of egg albumin has been extensively studied, and denaturation of soluble grain proteins has also been characterized. Determining the melting characteristics of fats is widely carried out. The method provides not only a precise melting point, but also a melting profile. The melting profile is related to the solid fat index (SFI) of the sample, but for a variety of reasons cannot be correlated directly to traditional SFI measurements. Freezing points, melting points and percent freeze-able water can all be measured with the DSC.

TGA, used less than DSC in the food industry, is a method that measures the weight change of a sample during heating or while holding at a constant temperature. Oxidation rates of oils at elevated temperatures can be measured as weight gain over time at a constant temperature. The reaction of the fat with oxygen results in a measurable weight gain. Rates of moisture loss at various temperatures during heating can provide insight into the free vs. bound water content of the sample.

MOISTURE SORPTION ISOTHERMS

Moisture Sorption Isotherms are plots of the equilibrium moisture content of a sample as a function of relative humidity. Samples of known initial moisture are equilibrated at several relative humidity levels. The weight gain or loss at each relative humidity is used to calculate the equilibrium moisture content of the product. The equilibrium moisture is then plotted against relative humidity to provide the graphic isotherm.

The information obtained from this analysis can be used to determine the compatibility during storage of different ingredients or components in a mixed system. The information is also used by packaging engineers to help define the moisture barrier requirements of the packaging needed for maximum shelf life of the product.

MISCELLANEOUS ASSAYS

The Brabender Amylograph and RVA described for wheat flour testing above also have application to other cereal grains, and are widely used in the starch industry. The method is particularly useful for measuring viscosity effects in modified and pregelatinized starches. Each processor tends to develop their own Amylograph and RVA methods, so a method reference is critical when analyzing starches.

Other methods utilized in Physical Testing include measurements of conductivity of liquids, specific gravity of liquids, and bulk and displacement densities of powders and particulate samples.

**A publication of Medallion Labs
Dr. Jonathan DeVries, Editor**

**If you would like to know more about Medallion Labs or would like to submit samples,
please visit our website at
www.medallionlabs.com or call us at 800.245.5615.**