

Measuring Cocoa, Chocolate Liquor and Products Using Microtrac Laser Diffraction Analysers

Introduction

One of the most enjoyable foods to consume is one made with a chocolate product. In the form of cakes, pies, candy bars, drinks and many other choices, chocolate morsels have been a choice for many years. The history of chocolate dates back to 4000 BC when cocoa was discovered in the Amazon. Subsequent to this, chocolate was used by the Mayan culture starting in 600 AD and eventually found its way into the Aztec culture in 1200 AD. Initially cocoa was used for preparing beverages and during conquest of part of Mexico in the 1500s. Hernando Cortez became interested in the use of cocoa as a form of currency. It may be his suggestion that the bitter drink when mixed with sugar, made the drink more palatable. The Spanish went further by mixing the beans with various ingredients including sugar, vanilla, nutmeg, and other flavourings to produce a food that was delicious, but only affordable by the wealthy. The ingredients were kept secret for 100 years by the Spanish.



Chocolate developed a reputation as an aphrodisiac during the reign of Louis XIV of France (1600s), which promoted the desire for chocolate. For approximately, the next 200 years chocolate was consumed as a drink. Finally, in the mid-1800s, chocolate as a solid was introduced while milk chocolate was introduced in the late 1800s. Since that time, chocolate has gained a reputation as a presentation of a show of love and admiration around the world. As part of this realization, chocolate has a wide appeal not only for those romantically inclined, but by people of all ages and cultures. It is an especial important part of the culture of many countries including Switzerland, Germany, France, Spain, Italy and America. The importance of chocolate

in the USA is clearly shown by the establishment of The Chocolate Manufacturers Association (CMA) of America in 1923. The photograph shows a cocoa pod containing beans.

As chocolate progressed in its stature as a desirable food, quality, consistency and regional specialization of flavour characteristics evolved. Part of the flavour profile of chocolate pertains to the particle size of the ingredients used to make the substance called chocolate. This paper describes particle size measurement of chocolate products or step products.

Processing

Chocolate processing has several basic steps, but variations exist depending on culture and population desires. The cocoa pod after harvesting from the cocoa tree, is fermented and dried, to produce what is known as the cocoa bean. The bean is then roasted by the chocolate manufacturer which causes the shell to separate to begin releasing the important nib. Isolation of the nib is accomplished by winnowing using a stream of air to effectively remove the shells before the next processing step. The nib of the bean is then ground. As with most grinding processes, the temperature of the mass increases resulting in a substance named chocolate liquor which contains 50-58% cocoa butter. Cocoa particles are also present having a size of approximately 100µm. The grinding and mixing action promotes the even distribution of cocoa butter throughout the mass causing the mix to start developing a creamy, smooth texture. The cocoa butter also contains the flavour ascribed to chocolate and cocoa products. Its release is the start of the development of the flavour profile of cocoa and chocolate. The chocolate liquor is then processed for use as cocoa powder (beverages and cooking) or chocolate. Unsweetened chocolate is chocolate liquor which has hardened. Cocoa is produced by a series of steps involving pressing the liquor which removes about one-half of the cocoa butter. The resulting cake from pressing is again ground to yield cocoa powder. The released cocoa butter can be used to aide chocolate manufacture by mixing with cocoa powder, sugar, flavours, milk, and other ingredients. In a different process, the nib is soaked in warm alkali, dried and then ground. This "Dutch" process produces a cocoa and chocolate that is much darker and redder. Dark chocolate is chocolate that does not contain milk solids (or very little) while milk chocolate is chocolate to which powdered or condensed milk is added. White chocolate does not contain cocoa products.

Chocolate is a special product having different processing steps than cocoa production in order to produce its special, flavourful properties. It is produced by mixing with other ingredients in a special step called conching (stirring in a steel vessel). This is the step that develops the final flavour profile of various chocolates of which "mouth feel" is one. Depending upon personal taste, chocolate can have a range of particle size distributions and flavour development. Conching causes the cocoa particles to be reduced to about 18µm from the original 100µm and requires anywhere from a few minutes to many days depending on the particle size distribution and general flavour profile desired. Normally, longer conching produces a more desired and higher quality chocolate. The final step in producing edible, quality chocolate is tempering wherein the chocolate is subjected to cycles of heating and cooling. During this process, small stable crystals of fat develop which promote a mouth feel of creaminess in addition to the particle size of ingredients

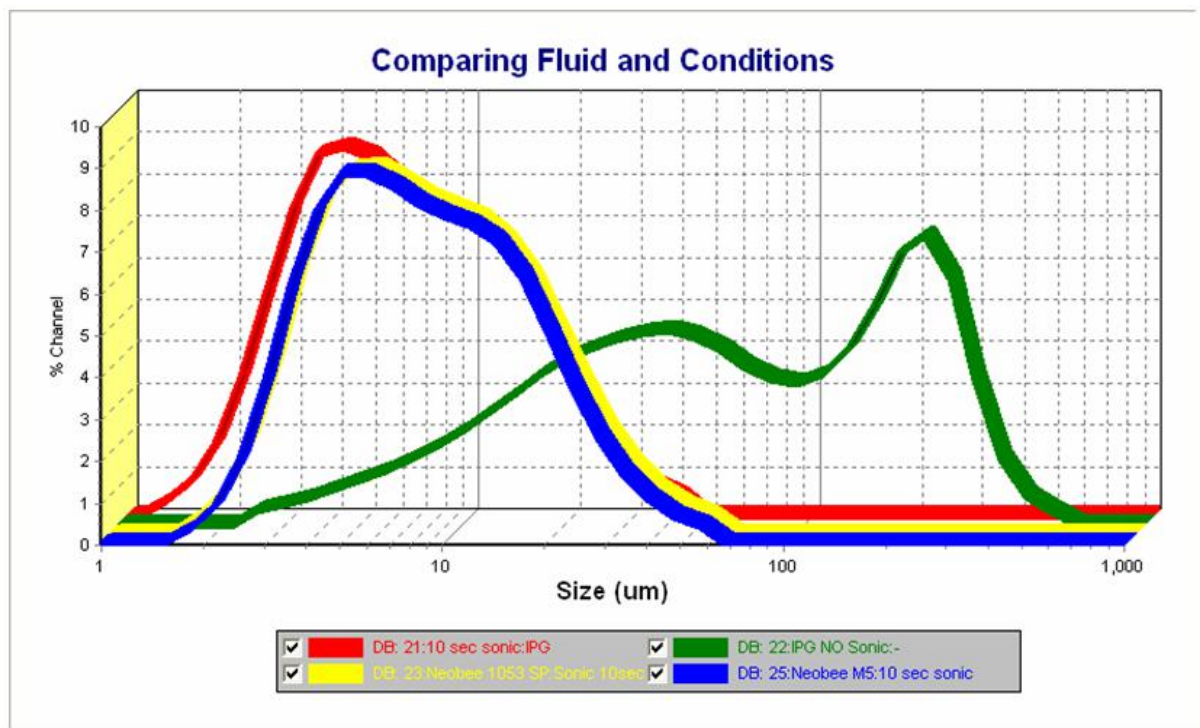
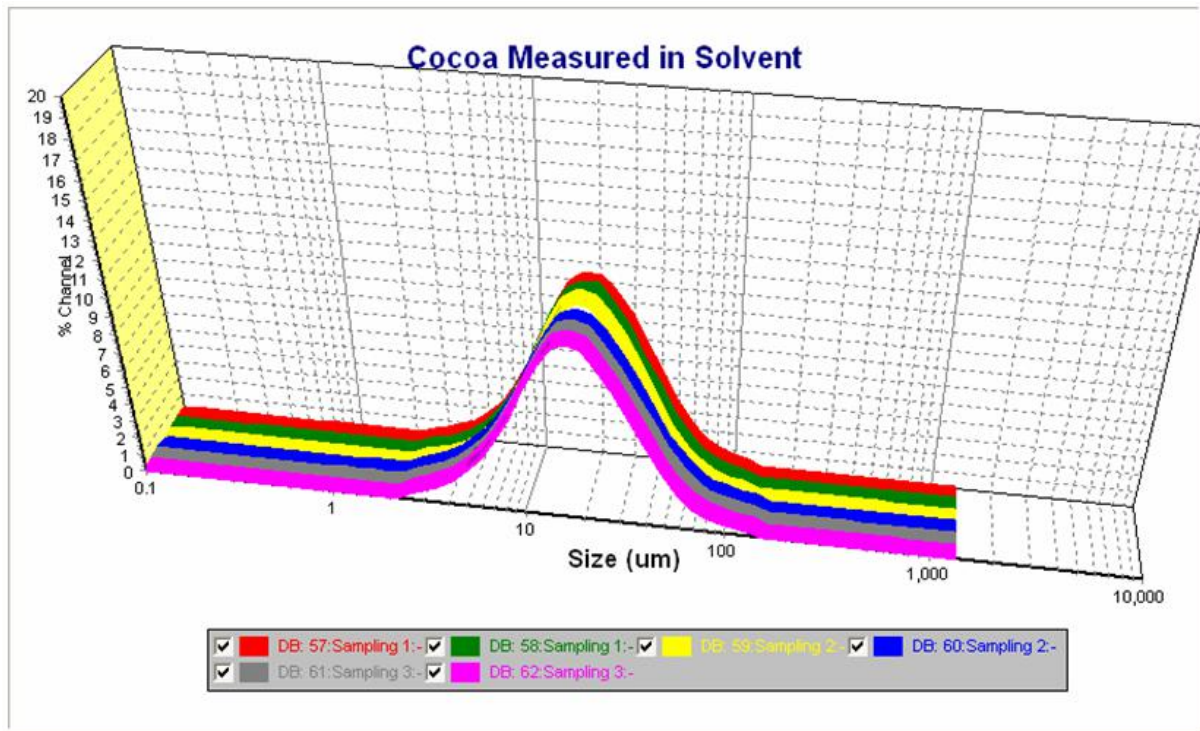
Particle Size Measurement

Many methods and technologies exist for measuring the size and distribution of cocoa and chocolate. These include sieves, optical and electronic particle counters, microscopy, sedimentation techniques, and laser diffraction instruments (the most used). Microtrac laser diffraction instruments have been used to measure particle distributions of chocolate products since 1976. Microtrac instruments continue to be used for this purpose using advanced concepts of optics and mathematics.

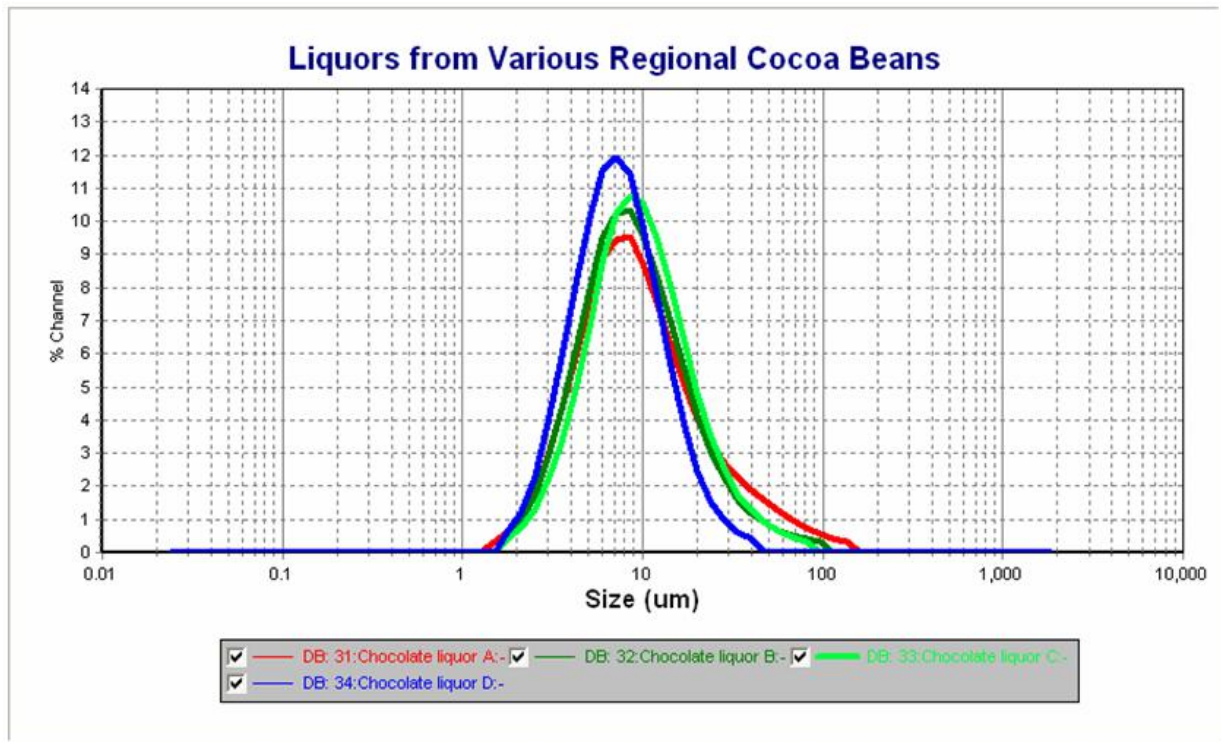
Cocoa product	Size and/or Range	Comment
Cocoa Particles Liquor	Median 50 – 100µm	
Cocoa Powder plus sugar	Smaller than 100 µm	If too small poor flow, requires more cocoa butter, chalky, sticky If too large, lack creaminess, gritty, poor distribution of cocoa butter
Conching	Start 100 µm, final 15 – 30 µm	
Dark Chocolate	Max size 65-75 µm	Small overall size than milk chocolate
Milk Chocolate	Maximum size 25 µ	Too coarse, slimy
Chocolate bar	Range 0.5 – 100 µm, D50 5 – 10 µm	
Cocoa, sugar, milk solids mix	Less than 30 or 25 µm according to some information	To assure blend is below the detectability limits of the tongue

Many fluids can be used for suspending and dispersing the particles contained within the various products. Many of the precursor materials can be dispersed without melting, but it has been found in our laboratory that chocolate products generally require the sample to be melted at approximately 55C to obtain a satisfactory sample for dispersion. While in the melted state, a portion of chocolate is transferred to the dispersing fluid to wet the particles. Ultrasonic energy is applied depending upon the results of microscopic examination. This examination allows a decision to be made to completely disperse the agglomerated particles. Note that dispersion by ultrasonic energy is not needed if agglomerates are to be identified as part of the particle size distribution. The fluids that may be used include Isopropyl alcohol, warm vegetable oil, silicone oil, higher purity organic oils, trichloroethylene, ethylene chloride, and high molecular, complex isoparaffins. The Microtrac Applications Lab located in Largo, Florida has examined these fluids over many years of measuring chocolate products. This paper will show the use of three of these fluids: Isopar G, Neobee 1053, and Neobee M-5 depending upon the experimental requirements.

In a reproducibility experiment, using cocoa as a base particle material, excellent precision was demonstrated. In this case IPG (Isopar G) was used. Each graph represents three separate samplings of the cocoa after suspension and dispersion. Also shown are duplicate measurements of the same circulating sample. Representative sampling techniques were used throughout the testing. In addition, the circulation system used in Microtrac instruments is especially designed to promote and maintain homogeneous suspension of particles necessary to obtaining accurate measurements of broad distributions.

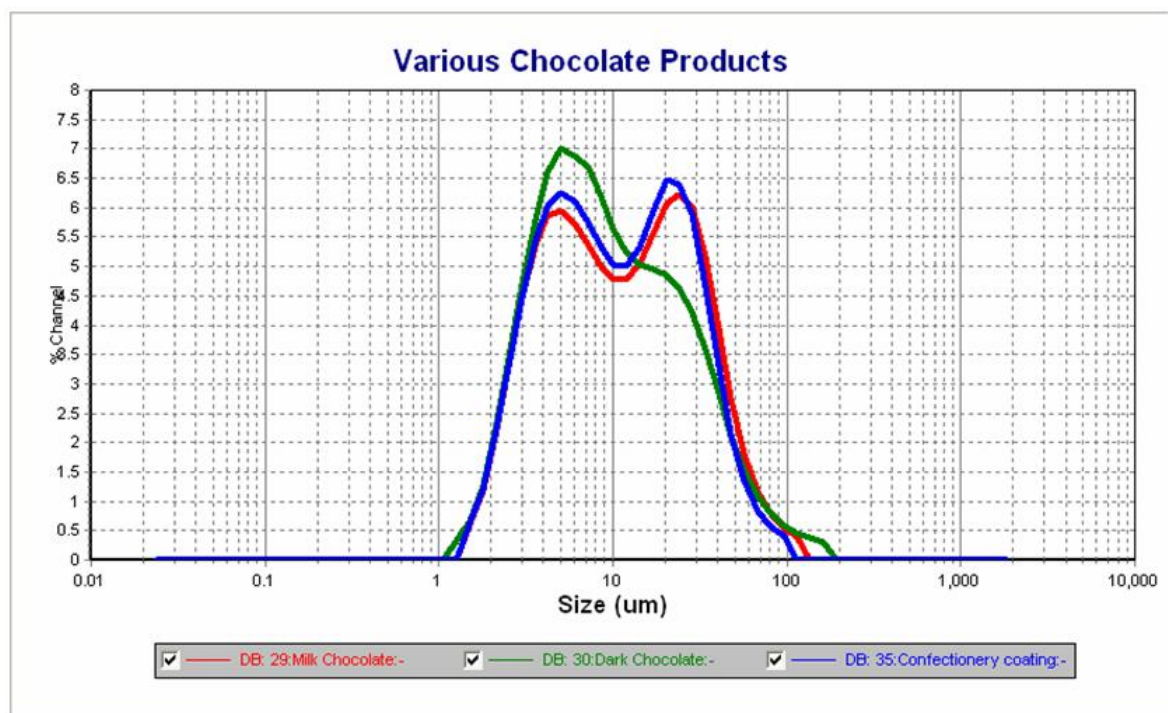


The graph above shows a comparison of using IPG (Isopar G/lecithin solution), NeoBee M5 and NeoBee 1053 as fluids for dispersal and operation when measuring a chocolate paste. Also present is the result of measuring sample in IPG without lecithin or ultrasonic energy. Lecithin acts as surfactant in organic media to allow better wetting and dispersion of particles in organic solvents that may show solubility in water or are difficult to disperse in other fluids. Without dispersion efforts of additional ultrasonic treatment, the particles remain undispersed and possibly trapped in a matrix of cocoa, sugar and fat. Of interest is the ability of IPG to provide better dispersion (smaller size) than the NeoBee oils.



Liquors can also be successfully measured using Isopar G. As shown above, chocolate liquors from several different global regions have different particle size distributions reflecting the different cultural desires in chocolate products.

Because of the different ingredients used to prepare dark chocolate, milk chocolate and confectionery chocolate, the particle size distribution of each can be expected to be different. The dark chocolate examined shows particle size as being smaller than the comparable milk chocolate and confectionery coating. Especially interesting is the bimodal nature of the distributions showing that more than one component is present in the mixtures.



Conclusions

Measuring Light scattering can be used for measuring the particle size during cocoa processing and chocolate manufacturing. Many fluids can be used including oils, organic solvents and silicone. For the experiments discussed above, Isopar G was used with lecithin as a surface active agent. Isopar G represents a fluid that must be disposed by special means, while NeoBee is a GRAS substance that can be handled safely, stored, reprocessed for other use or discarded as part of animal feed. Depending upon the situation in a particular laboratory setting, either of the materials used for this article or others can be used to suspend particles. Isopar G and isopropyl alcohol are easiest to clean should a spill occur. Silicone oils usually lend themselves to cleaning with alcohols but also require special disposal methods and leave residue. Vegetable or other food or synthetic food oils can be cleaned easily with soaps. Regardless of the choice of fluid, Microtrac diffraction instruments can accept the fluid of choice due to its design features without changing tubing, pump parts or other manipulation. One example for such an instrument will be the **Microtrac MRB Sync**.



References:

1. Chocolate, Cocoa and Confectionery: Science and Technology, 1980 Bernard W. Minifie
2. Chocolate history timeline: www.chocolate-monthclub.com
3. Methods for measuring particle size distribution of chocolate products. 37th PMCA Production Conference.
4. Photo: Chocolate Manufacturers Assoc. www.cocoatree.org