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List of Acronyms

None

Executive Summary

The United States Department of Energy (DOE) Advanced Manufacturing Office (AMO) award (DE-EE0005774) provided support for the development of emulsion, atomization and dryer controls technologies to address unique technical issues associated with high production implementation of the associated drying technology. The typical spray drying process used today employs air heated up to 400° Fahrenheit to dry an atomized liquid into a powder. ZoomEssence technology uses no air heating, delivering higher quality flavors and ingredients with longer shelf life and improved solubility. The scope of this project was to develop (1) improved ability to formulate emulsions for specific flavor groups and improved understanding of the relationship of emulsion properties to dry particle properties, (2) atomizer(s) with increased production rate and (3) a data acquisition system.

Industrial spray drying is an extremely energy intensive process consuming between 10 and 20 % of total industrial energy use in most industrialized countries.(Mujumdar 2011) The most common approaches to improving efficiency are 1) reducing the evaporative load 2) improving heat recovery or insulating the process system and 3) increasing the efficiency of heat supply (boiler system, etc.). Altogether, these approaches to increasing efficiency do not address the fundamental cause of inefficiency: *high temperature processing*. In addition to decreased efficiency, conventional spray dry systems result in significant product degradation. (Piatkowski 2003) Products dried at high temperatures can go through chemical and physical conversions significantly reducing their sensory appeal and stability. Nutraceuticals can lose their potency due to high temperature oxidation and degradation. (Mujumdar 2011) The ambient drying temperature of the DriZoom™ process preserves the chemically active and highly volatile compounds in flavors, fragrances, pharmaceuticals, and chemicals while consuming less than half of the energy costs of a traditional spray dryer. (see benefit analysis on page 15)

Investigations into Task 1: Emulsion Formulation resulted in the development of “emulsion templates” which are flavor dependent classes of emulsion formulations. The different classes retain consistency of emulsion properties such as viscosity, stability and emulsified oil droplet size distribution between the different classes of products dried. Task 2: Atomizer Development resulted in the design of 2 production atomizers and 1 back-up atomizer. Research and development into the atomizer indicates greater control over particle size, more homogeneous particle morphologies and potential for greater dryer throughput. Task 3: Dryer Control System focused on the development of a data acquisition system to meter and optimize the use of energy throughout the drying process. Temperature, pressure, flow rate and relative humidity measurements were placed in critical locations throughout the production process to acquire data for use in optimization of dryer parameters.

Learnings in these areas are being applied for optimization of particle formation

and drying. Formulation and atomization improvements led to significant increase in dryer throughput. These and future learnings will lead to significant improvements in efficiency and capacity as throughput is increased using current infrastructure. Current work is now focusing on use of dryer control data for measurement and improvement of system energy uses.

Introduction

ZoomEssence developed its patented DriZoom^{TM} technology that converts liquids to powders at ambient temperature. The fundamental idea of spray drying is the production of highly dispersed powders from an atomized fluid feed by evaporating the solvent. The process of drying a liquid into a powder form has been traditionally achieved by mixing a heated gas with an atomized (sprayed) fluid within a vessel (drying chamber) causing the solvent to evaporate. The typical spray drying process used today employs air heated up to 400° Fahrenheit to dry an atomized liquid into a powder. Exposing sensitive, volatile liquid ingredients to high temperatures causes molecular degradation that negatively affects solubility, stability and flavor profile of the powder. In short, heat is detrimental to many high-value liquid ingredients. Spray drying has become the most important method for the dehydration of fluid foods such as milk, coffee and egg powder, and is used extensively in the food, pharmaceutical and chemical industries.

ZoomEssence's patented technology (Beetz et.al.) fundamentally changes the drying process by eliminating the use of heat. Because of our unique process ZoomEssence delivers higher quality flavors and ingredients, enables longer shelf life and promotes improved solubility. It was also discovered that the ability to manufacture dry particles at ambient temperature supports a higher yield in the manufacturing process. This "green" approach will save significant energy and permits the design of a new and a much less expensive dryer than current systems. The DriZoom^{TM} technology can be applied in a variety of processes used to create and deliver flavor, fragrance, ingredient, chemical and pharmaceutical powdered products.

Presently ZoomEssence has developed a 'no-heat' dryer. The outcome of this program will be (1) improved ability to formulate emulsions for specific flavor groups and improved understanding of the relationship of emulsion properties to final dry particle properties, (2) atomizer(s) with increased production rate and (3) a data acquisition system.

Background

Traditional spray drying technology dates back to the 1870's with the drying of agricultural products. Between 1870 and 2008, the fundamentals of spray drying have stayed consistent: a liquid slurry is atomized into a hot stream of gas, where water and other volatile content is vaporized and the dry product is collected. At the core of spray drying is the atomization technology used, which controls how the liquid is dispersed into droplets before drying. (R.P. Patel 2009)

To get air temperatures hot enough for traditional spray drying, either gas or coal fired boilers are used to produce steam for heat exchangers to supply dryers with hot air. In some particular non-food applications, combustion gases directly from natural gas combustion are used to heat the air for the drying process. Due to both the prevalence of spray drying as a method to convert liquids to solids and inefficiencies associated with running any high temperature process, spray drying consumes approximately 10% – 20% of energy within the industrial sector. (Mujumdar 2011)

Due to the high temperature (> 200°C) of traditional spray dryers, drying is broken down into multiple phases. After atomization, the surface temperature of spray dried particles rapidly increases due to the high temperature of the ambient air. As the temperature approaches the boiling point of water, the surface of the particle begins to boil and dehydrate, thus resulting in a thick film surrounding the outside of the particle. (Kim, Chen et al. 2009) As liquid water begins to turn into vapor within the interior of the particle, the thick film on the exterior of the particle prevents diffusion. Therefore, a vapor bubble forms in the interior of the particle, resulting in a hollow particle surrounded by a thin external shell of flavor droplets.(M. Rosenberg 2016) Depending on the temperature of drying, particles can rupture due to the vaporization, releasing the pressurized gas inside.(Fang, Rogers et al. 2012) Water removal from particles in the DriZoom™ process takes place in a much different manner, as particle temperatures stay far from the boiling point of water.

The objective of this DOE AMO award is to address unique considerations associated with the "no heat" process through developments in 1) emulsion formulation, 2) atomization and 3) data acquisition systems. Understanding the way different flavor compounds impact emulsion rheology is critical for streamlining emulsion properties for potential end users. Atomization is a necessary component of any spray drying application. Typical spray dry applications atomize liquids with viscosities of 300 – 500 centipoise. DriZoom™ emulsions are much higher, requiring a different approach to reliably produce uniform, spherical particles.

Standard spray drying operations measure efficiency as $E = 100 \times (T_{in} - T_{out}) / (T_{in} - T_{ambient})$, which interprets the passing of energy from the heated air inlet into the product. This efficiency measurement does not take into account other drying modes outside of the enthalpy of vaporization of water and heat capacity of air.

Considering the DriZoom™ process is not reliant on thermal input for drying, this efficiency measurement is rendered moot. In order to measure and optimize the efficiency of the DriZoom™ technology, a data acquisition panel is required to measure all process inputs (feed rates, temperatures, pressures etc.) to measure and optimize the efficiency of the DriZoom™ process.

Results and Discussion

Task 1: Improvements in Emulsion Formulation

The objective of Task 1 was the development of emulsion and particle characterization functions that provide information on the particle – flavor interactions. Connections were made between ingredients used for specific flavor types and their impacts on viscosity, emulsion stability and dry powder performance. Additionally, this information has been used to expand the range of products to dry and aided in formulation of high load formulas which reduce drying time and increase overall capacity.

Subtask 1.1: Correlation of Emulsion and Dry Particle Size

Emulsion Formation

Emulsion particle size refers to the size of the oil / flavor droplet diameter within the slurry. Small diameter droplets are preferred as they produce stable emulsions. Larger oil droplet diameters are unstable. As part of the preliminary investigation to understand the formation of stable emulsions, oil droplet diameters were evaluated as a function of processing time.

Single Molecule Studies

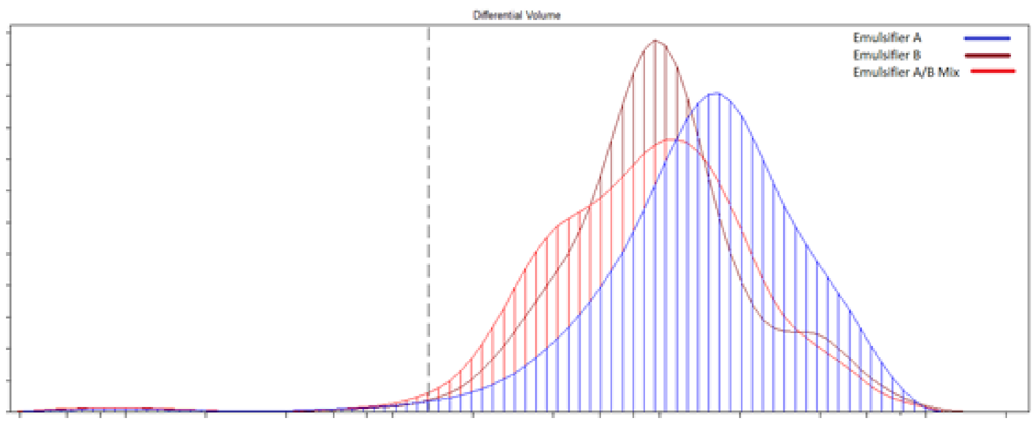
Understanding the interaction between the oil being emulsified and the emulsifier is critical for development of stable emulsions. In this regard, a system of single molecule studies were performed to understand the impact of oil type on emulsion stability, oil droplet diameter, viscosity and particle size. Emulsion and dry particle properties were investigated as a function of single molecule content.

Dry particle sizes were captured for all materials. Between drying the different materials, all dryer properties (including atomization speed and rate, dryer temperature etc.) were set the same.

Organic Fruit

After a basis for appropriate liquid and particle size distribution and impact of manufacturing process was understood on products, new emulsification systems were evaluated to address end-user requirements. Three emulsifiers A, B and A/B mixes were evaluated. Emulsifier A resulted in a stabilized emulsion of moderate to high viscosity. Additionally, excess Emulsifier A was required during formulation to achieve a stable emulsion, leading to higher viscosities. On the other hand, Emulsifier B significantly reduces viscosities of resultant emulsions. Emulsifier B is also required in less quantity as stable emulsions were formed at low Emulsifier B concentrations. A combination of emulsifier A and B demonstrated the ability to form a stable emulsion while controlling the viscoelastic properties of the emulsion.

Dry particle size measurements were used to evaluate the performance of spray dried emulsions. All particle size distributions displayed significant distribution overlap, which is a function of atomizer design, speed and emulsion feed rate. Future studies in Task 2 look to develop correlations on this portion of the atomization front. In conclusion, successful formulations were developed for high viscosity emulsions on organic carriers, addressing a consumer need for organic based formulas.



Subtask 1.1: Conclusion
Significant advancements have been made in the formulation of low water content, high viscosity emulsions used in the DriZoom™ formulation process. A series of emulsifiers and bases have been identified and employed for the formulation of low viscosity emulsions with stable oil droplet diameters. Emulsion preparation is now a commercial process, with bench formulation matching pilot and production preparation with predictable scalability. Particle diameters are consistent and dictated by both the emulsion and atomization technique. Work in Task 2 will develop the atomization technology and develop and understanding of particle formation.

<u>Subtask 1.2: Improvement in Thermal Stability of Powders</u>

Glass Transition Temperature

The glass transition temperature of spray dried powders is the temperature at which the powder transforms from an amorphous powder to a rubbery state. In the rubbery state, particles begin to fuse together, referred to as "caking". Caking of materials negatively impacts performance, as the material is no longer a free flowing powder and is difficult to handle and solubilize. The objective of this work is to create formulations which increase the glass transition temperatures of the final spray dried powders.

The dynamic mechanical analyzer (DMA) acquired measures the strain of a material as a function of an applied sinusoidal stress. From this, the complex modulus of the material can be calculated and subsequently the storage and loss modulus. The glass transition temperature is determined from measuring the storage modulus as a function of temperature and looking for the temperature at which there is a sharp decline in the storage modulus of the material.

Subtask 1.2: Conclusion

The DMA has been a critical piece of equipment in determining the cause of clumping and bricking of powder products after manufacture. Substantial improvements have been made in our emulsion formulation process to resolve potential sources of bricking before they become problematic.

Impact:

Control over emulsion performance characteristics has resulted in increased up-time in production. Formulations have been modified to prevent caking. In combination with the control system of Task 3, up-time, down-time and run times will be correlated with run rates, atomizers and emulsions to optimize the DriZoom™ process. Multiple variables from formulation to atomization and tank conditions were used to increase capacity and dryer throughput in Task 2 and 3.

Task 2: Continued Atomizer Development

The objective of this task was to develop an atomizer capable of increasing both throughput and the desired particle size. This trial atomizer was then placed in manufacturing for testing and evaluation, where it demonstrated successful atomization with significant throughput increases. This task is culminating in the design of 2 production atomizers and 1 back-up atomizer, and successfully achieved [the targets set forth above].

Task 3: Data Acquisition Panel

After the engineering design phase, an installer was selected to implement the dryer control plan. The engineering phase designed the location and arrangement of various sensors throughout the manufacturing process. After installation and tuning of the panel, data is outputting from control points properly. Pressure and temperature readings throughout the DriZoom™ system have been used to better understand system performance. Dewpoint measurement readings across the system indicate room for increased capacity. Dryer optimization runs estimate capacity increases up to 65%.

Benefits Assessment

A key goal of this DOE – Industry partnership is to demonstrate how government resources can be used to support U.S. industry in developing highly energy efficient spray dried powder manufacturing technology. Based on evaporation requirements alone, Zoom's process result in an estimated reduction of the energy requirement by a factor of two = 2.4 GJ / ton energy savings. This gives an estimated global energy savings on the order of 240,000 GJ just in spray dried food products. This work also impacts a wide array of more traditional drying processes beyond spray drying.

Optimization of formulation and atomization have led to larger throughputs and increased dryer capacity. Prior to the DOE investment, approximately 3688.7 BTU / lb of dried product was used. The new generation atomizers developed through this award have demonstrated alone the ability to increase throughput by up to 65%. Improvements in emulsion formulation have demonstrated the ability to deliver double the concentration of actives per pound of powder, reducing overall use rates and increasing drying capacity two fold. A balanced approach between process capacity and formulation capacity has been implemented in capacity expansion. Therefore, by throughput increases alone, power requirement energy requirements have been reduced to 2235 BTU / lb product. Initially a factor of 10 was applied to sizing dehumidifiers for protoype dryers. It has been determined this factor is significantly higher than required. In the future, a 2 to 3 fold reduction in dehumidifier sizing will be applied, thus reducing the energy required for DriZoom™ product between approximately 800 and 1100 BTU / lb finished product.

A recent report by APV (APV Dryer Handbook, Invensys Tonawanda NY), a typical large capacity spray dryer operates at approximately 1151.3 BTU / lb product dried. A critical consideration must be made between DriZoom™ and Spray Dry products: use rates. Use rates of DriZoom™ powder are typically 2-3 fold lower compared to typical spray dry powders due to significantly increased actives retention in DriZoom™ processing. Therefore, in use, spray dry products require approximately 3454 BTU / lb of flavored finished product. A smiliar U.K. government report determined 2307 BTU / lb finish product as well for traditional technologies. Therefore, on an energy cost per pound (BTU / lb), traditional spray dry is estimated between 3400 and 4600 BTU / lb in finished product. This is 3-5.75 fold higher than the forecasted 800 to 1100 BTU / lb of the DriZoom™ process.

Significant improvement in energy efficiency can also be gained through further process development and scale. The current spray dry flavor market is approximately 500 million pounds of flavor per year. ZoomEssence intends to increase its current dryer capacity by a factor of at least 10 times within the next 10 years. With this increase in market share, industry wide energy savings will be seen through implementation and efficiency gains in DriZoom™ technology.

Commercialization

To displace current suppliers, ZoomEssence offers significant advantages in product quality and cost to entice customers to utilize our product. Our value proposition of liquid quality flavors and ingredients in a powder form had not been previously achieved in the spray drying industry. Through the first 9 months of 2015, 130 new formulas were made with 86 new commercializations. ZoomEssence will use existing distribution channels to grow business with continued focus on high-value flavor products. The DriZoom™ product currently outperforms traditional products due to flavor retention in the finished powders. Significant use rate reductions of 30% or more are frequently seen by switching to DriZoom™ flavors. Future growth plans focus in the flavor and specialty ingredient markets using existing industry distribution channels.

Conclusions

In regards to process development, ZoomEssence has integrated the research equipment acquired into its new product development protocols to ensure quality, and increase manufacturing capability and reproducibility of its products. The particle sizer, specifically, has been used not only as a quality check, but also in atomizer development. The Dynamic Mechanical Analyzer aids in new material resourcing and ensures product stability. The installed data acquisition panel has quickly become a much valued resource. In the event of manufacturing difficulties, process data is immediately consulted to resolve any discrepancies in manufacturing.

ZoomEssence's research in 1) emulsion development, 2) atomizer development and 3) data acquisition have substantially progressed ZoomEssence's capabilities. Product development can now be approached quantitatively with the R&D equipment acquired. Atomizer development efforts have resulted in consistent and reliable atomizers. The data acquisition panel has produced a starting point for process development and optimization of our energy efficiency and process technology.

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