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14. ABSTRACT The purpose of this project was to establish state-of-the art facilities to develop nanoparticle and microparticle encapsulation systems for bioactive components that may improve soldier performance, e.g., nutrient delivery systems or antimicrobial delivery systems. As part of the project we purchased facilities to fabricate nanoparticles (spray drying unit) and equipment to characterize the powders produced (freeze drying, sputter coating, and scanning electron microscopy). During the course of the project we have developed a wide range of nanoparticle and microparticle delivery systems that can be used to encapsulate, protect, and release bioactive components.					
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Report Title

Final Report: Bioactive Encapsulation for Military Food Applications: Request for Enhanced Nano and Micro Particle Fabrication and Characterization Facilities

ABSTRACT

The purpose of this project was to establish state-of-the art facilities to develop nanoparticle and microparticle encapsulation systems for bioactive components that may improve soldier performance, e.g., nutrient delivery systems or antimicrobial delivery systems. As part of the project we purchased facilities to fabricate nanoparticles (spray drying unit) and equipment to characterize the powders produced (freeze drying, sputter coating, and scanning electron microscopy). During the course of the project we have developed a wide range of nanoparticle and microparticle delivery systems that can be used to encapsulate, protect, and release bioactive components, including biopolymer complexes, protein nanoparticles, lipid nanoparticles, and microgels. For example, we have encapsulated natural antimicrobials (such as essential oils and polylysine), vitamins (vitamin A, D, and E), and nutraceuticals (curcumin, betacarotene, w-3 fatty acids, polyphenols, and Coenzyme Q10). Vitamins and nutraceuticals may promote soldier performance, whereas antimicrobials may be used to improve the shelf life and safety of rations.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Book

TOTAL:

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

NAME

PERCENT SUPPORTED

FTE Equivalent:

Total Number:

Names of Post Doctorates

NAME

PERCENT SUPPORTED

FTE Equivalent:

Total Number:

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period:

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:.....

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:.....

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):.....

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:.....

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:.....

Names of Personnel receiving masters degrees

<u>NAME</u>
Total Number:

Names of personnel receiving PHDs

<u>NAME</u>
Total Number:

Names of other research staff

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Technology Transfer

Equipment was purchased.

Bioactive Encapsulation for Military Food Applications: Request for Enhanced Nano- & Micro-particle Fabrication & Characterization Facilities

Introduction

The purpose of this project was to establish state-of-the-art facilities to develop nanoparticle and microparticle encapsulation systems for bioactive components that may improve soldier performance, e.g., nutrient delivery systems or antimicrobial delivery systems. As part of the project we purchased facilities to fabricate nanoparticles (spray drying unit) and equipment to characterize the powders produced (freeze drying, sputter coating, and scanning electron microscopy). During the course of the project we have developed a wide range of nanoparticle and microparticle delivery systems that can be used to encapsulate, protect, and release bioactive components, including biopolymer complexes, protein nanoparticles, lipid nanoparticles, and microgels. For example, we have encapsulated natural antimicrobials (such as essential oils and polylysine), vitamins (vitamin A, D, and E), and nutraceuticals (curcumin, beta-carotene, w-3 fatty acids, polyphenols, and Coenzyme Q10). Vitamins and nutraceuticals may promote soldier performance, whereas antimicrobials may be used to improve the shelf life and safety of rations.

The physicochemical properties of nanoparticle and microparticle encapsulation systems have been characterized, including particle size distribution, electrical charge, and morphology. In addition, the biological activity of antimicrobials has been established using model microorganisms, whereas the potential bioavailability of nutraceuticals has been established using a simulated gastrointestinal tract (GIT) model that includes mouth, stomach, and small intestine phases.

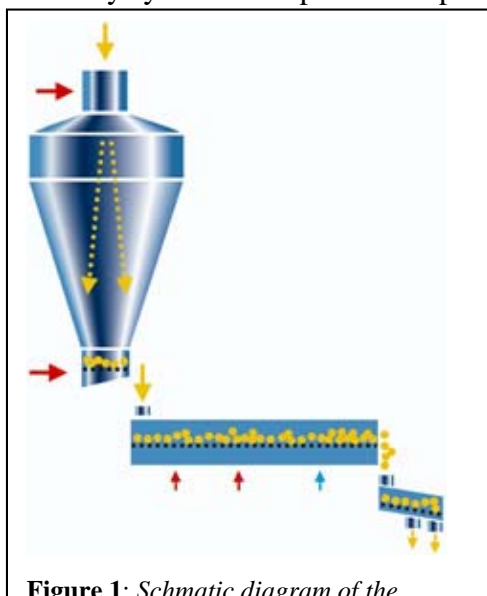


Figure 1: Schematic diagram of the principles of a small scale spray drier that can be used to convert fluid colloidal delivery systems into agglomerated powders (from Niro.com).

Particle Fabrication Facilities

Spray drier – A small-scale spray drier was purchased to convert fluid delivery systems into solid powders using *microencapsulation*. Microencapsulation involves passing a fluid through a tiny nozzle to form a mist of small drops, which are then dried by evaporating the solvent at elevated temperatures (**Figure 1**). This unit has been used for drying delivery systems. The instrument purchased mimics the drying conditions that occur within industrial scale spray driers, so as to facilitate scale-up from pilot plant to production. The advantage of purchasing a small-scale spray drier is that it can

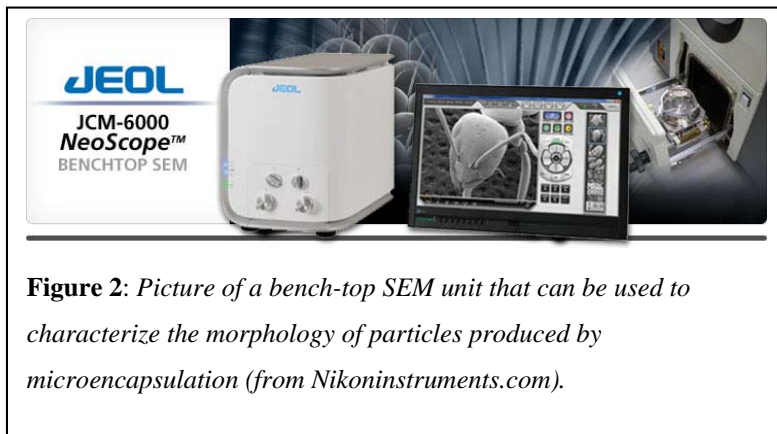


Figure 2: Picture of a bench-top SEM unit that can be used to characterize the morphology of particles produced by microencapsulation (from Nikoninstruments.com).

be utilized to rapidly screen the efficacy of different construction materials and structural organization principles used in developing delivery systems. This has provided us with the ability to test different wall materials in the powders that can protect the nutraceuticals during storage by inhibiting oxygen and light exposure, while also enhancing the bioaccessibility of the microencapsulated nutraceutical after they are dispersed in foods and beverages.

Particle Characterization Facilities

Bench-top SEM – A bench-top scanning electron microscope (SEM) was purchased to characterize the morphology and dimensions of the structured delivery systems prepared using the microencapsulation method. This instrument enables the microstructure of materials to be determined by passing an electron beam across their surface (**Figure2**). The functional performance of bioactive components is known to be strongly influenced by their nano- and micro-structure, and so knowledge of their morphology is essential for understanding and controlling their stability and properties.

Applications

The equipment is being used to educate students on the fabrication and characterization of nanoparticle and microparticle delivery systems, as well in fundamental research aimed at designing delivery systems to: improve the stability of bioactive agents in food products/rations; their dispersibility in food products and rations; and enhance their biological activity (such as antimicrobial activity or bioavailability).

Representative Publications

Komaiko, J., & McClements, D. J. (2014). Optimization of isothermal low-energy nanoemulsion formation: Hydrocarbon oil, non-ionic surfactant, and water systems. *Journal of Colloid and Interface Science*, 425, 59-66.

Komaiko, J., & McClements, D. J. (2015c). Low-energy formation of edible nanoemulsions by spontaneous emulsification: Factors influencing particle size. *Journal of Food Engineering*, 146, 122-128.

Landry, K. S., Chang, Y., McClements, D. J., & McLandsborough, L. (2014). Effectiveness of a novel spontaneous carvacrol nanoemulsion against *Salmonella enterica* Enteritidis and *Escherichia coli* O157:H7 on, contaminated mung bean and alfalfa seeds. *International Journal of Food Microbiology*, 187, 15-21.

Lopez-Pena, C. L., & McClements, D. J. (2014a). Optimizing delivery systems for cationic biopolymers: Competitive interactions of cationic polylysine with anionic K-carrageenan and pectin. *Food Chemistry*, 153, 9-14.

Lopez-Pena, C. L., & McClements, D. J. (2014c). Optimizing delivery systems for cationic biopolymers: Competitive interactions of cationic polylysine with anionic kappa-carrageenan and pectin. *Abstracts of Papers of the American Chemical Society*, 247.

Ozturk, B., Argin, S., Ozilgen, M., & McClements, D. J. (2014). Formation and stabilization of nanoemulsion-based vitamin E delivery systems using natural surfactants: Quillaja saponin and lecithin. *Journal of Food Engineering*, 142, 57-63.

Saberi, A. H., Fang, Y., & McClements, D. J. (2014a). Effect of Salts on Formation and Stability of Vitamin E-Enriched Mini-emulsions Produced by Spontaneous Emulsification. *Journal of Agricultural and Food Chemistry*, 62 (46), 11246-11253.

Saberi, A. H., Fang, Y., & McClements, D. J. (2014b). Stabilization of vitamin E-enriched mini-emulsions: Influence of organic and aqueous phase compositions. *Colloids and Surfaces a-Physicochemical and Engineering Aspects*, 449, 65-73.

Saberi, A. H., Fang, Y., & McClements, D. J. (2014c). Stabilization of Vitamin E-Enriched Nanoemulsions: Influence of Post-Homogenization Cosurfactant Addition. *Journal of Agricultural and Food Chemistry*, 62 (7), 1625-1633.

Yao, M., Xiao, H., & McClements, D. J. (2014). Delivery of Lipophilic Bioactives: Assembly, Disassembly, and Reassembly of Lipid Nanoparticles. *Annual Review of Food Science and Technology*, Vol 5, 5, 53-81.

Yi, J., Zhu, Z., McClements, D. J., & Decker, E. A. (2014). Influence of Aqueous Phase Emulsifiers on Lipid Oxidation in Water-in-Walnut Oil Emulsions. *Journal of Agricultural and Food Chemistry*, 62 (9), 2104-2111.

Zhang, Z., Decker, E. A., & McClements, D. J. (2014). Encapsulation, protection, and release of polyunsaturated lipids using biopolymer-based hydrogel particles. *Food Research International*, 64, 520-526.

Zheng, J., Li, Y., Song, M., Fang, X., Cao, Y., McClements, D. J., & Xiao, H. (2014). Improving intracellular uptake of 5-demethyltangeretin by food grade nanoemulsions. *Food Research International*, 62, 98-103.