Cover Page

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Development of Soft Matter Wet Lab and Analytical Capability in LEO Destination

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Background

Soft matter is the field of science that generally deals with "squishy" materials, in particular non-Newtonian fluids. Examples of soft matter include colloids, polymers, foams, gels, granular materials, liquid crystals etc. In the recently concluded Grand Challenge Workshop [1], study of non-equilibrium phenomena from macro- to nano-scale for soft matter was identified as the future focus area for NASA. Three research themes were identified as key to achieving success in the focus area – (a) smart reactive materials and systems, (b) self-reliant sustainable ecosystem and (c) active materials and metamaterials. In the recently concluded call for topical white papers, researchers submitted white papers under one or multiple of these themes. One of the key platforms for conducting microgravity research in the field of soft matter is LEO facility. Presently, ISS is the only LEO facility. However, in the coming decade, there is expected to be multiple commercial LEO destinations. While this white paper focuses on "soft" materials, many if not all of the suggested analytical techniques have multi-purpose capabilities across wide range of scientific domains.

Challenges with Soft Matter Research in ISS

In the last two decades, there has been significant amount of research in the field of soft matter in ISS. The present protocol of research requires pre-packaging of samples well in advance on ground and then launching it ISS. The cost of each such iteration ~ USD 250K- 2 million. Each iteration takes ~ 2-4 years. If further studies are to be executed on the samples and they still exist on station, the delay is perhaps only days or weeks and the bulk of the costs have already been contributed. This impacts soft matter research in multiple ways. The long duration between experiments reduces the number of experiments that can be executed on ISS. The high cost of sample preparation and launch makes it cost prohibitive to execute multiple iterations. These in turn prohibits opportunity to test out multiple ideas and forces NASA and CASIS to focus on only very few flight programs. Also, the long time required prior to launch also prohibits us from executing relevant non-equilibrium research, as identified by the Grand Challenge Workshop. Additionally, the analytical capabilities on station are limited, minimally effective or not working.

Plans to Improve Soft Matter Research in ISS and Future LEO Destinations

As identified in the challenges, described above, the way to improve the efficiency of soft matter research in LEO destinations, two most important criteria that needs to be fulfilled are-

(a) Ability to produce samples for soft matter research on LEO destination aka *"Wet Lab"*
(b) Improved analytical capabilities to characterize and study the as produced soft mater.

The proposed wet lab and analytical capabilities will have the following objectives:

1. Develop a plan for the implementation of a suite of analytical technologies on the International Space Station for use by NASA and the ISS US National Lab.
2. Acquisition of the technologies selected and ready them for use on station.
3. Integration of the analytical tools on station and verification of the as prepared samples.
4. Development and implementation of the data systems that record, hold and provide access to the data that is produced.

Wet Lab. A wet lab is defined as the lab facility that will have the capability to handle soft matter. Some of the key requirements for wet labs are-

- ✓ Ability to contain sample during preparation and ability to seal the sample
- ✓ Ability to handle wide range of non-Newtonian fluids

- ✓ Ability to produce sample with and without the intervention of crew
    - o Robotic arm-based capability that can be controlled from ground

Analytical Capability. As mentioned earlier, presently ISS has very limited analytical capability with regard to soft matter research. Here a list of some of the new analytical capabilities and brief background is provided. We would like to mention that desired analytical capability isn't limited to the mentioned instrumentation and can be augmented based on the feedback from the soft matter community.

<u>FT-IR</u>

Fourier-transform infrared spectroscopy (FT-IR spectroscopy) measures the vibrational frequency of chemical bonds using infrared light. The wavelength of infrared light absorbed is directly related to the energy of each unique bond. As such, recording the IR absorbance over a range of wavelengths provides a spectrum that is unique to that molecule. IR spectroscopy observes chemical bonds which undergo a change in their dipole moment upon excitation with IR light. Modern IR systems come with- **Libraries** and **Software** that helps the user to take a spectrum, capture the output and includes Multi-Component Search (MCS) algorithms to identify the components of mixtures with better accuracy than search and subtract methods. Technology that comes with factory-verified specifications such as signal-to-noise ratio (50,000:1) and spectral resolution (0.25 cm^{-1}) By adding components to the system upgrades can be built into the same platform allowing for us to integrate the system into a flow reactor, probe batch processes from drop size to multi kilo scale and to conduct failure analysis and characterize unknowns by adding TGA-IR or FTIR microscopy. Many modern FT-IR systems are compatible with hundreds of accessories. For the experiments with regard to soft matter, the vibrational spectrometer will need to have a high signal to noise of at least (10,000:1). This should be a very straightforward modular technique to adapt to the automated flow chemistry systems that have already been developed for ISS-NL use.

<u>Raman Spectroscopy</u>

Raman spectroscopy provides information about the vibrational modes of compounds like FT-IR, but is mechanistically a different phenomenon. A Raman spectrum records the wavelengths of IR light which are scattered as a result of change in polarizability of a chemical bond. IR and Raman provide complementary information about the vibrational characteristics of molecules. Raman spectroscopy can be used to characterize molecules which cannot be observed by conventional IR. In-flow Raman has the same advantages as in-flow IR, and can be used for similar purposes. This is a non-destructive technique and would be a compliment to IR for the analysis of solution reaction chemistries for soft matter, which also include solid and semi-solid structures.

<u>Fluorescence Spectroscopy</u>

Fluorescence is the phenomenon where a molecule absorbs light within its absorption band and then emits this light at longer wavelengths within its emission band. This phenomenon can be used to identify, quantify, and observe chemical activity, and it is a popular method due to its high levels of sensitivity, simplicity, and specificity. This tool has applications for systems in biomolecules, small organic and inorganic molecules and materials. We are looking for system with Fluorescence excitation (and emission) in the range of at least 200-800 nm and resolution of +/- 1 nm (for wavelength). Fluorescence spectroscopy is a versatile and well understood analytical tool that can be used for biological systems and chemical analysis of solutions, mixtures and pure materials. It is non-destructive (does not destroy or modify sample being analyzed) and can be built into a dual system that would provide both fluorescence and Raman spectroscopic capabilities. As part of this effort, we will look at delivering a fluorescence system that could be used for other purposes on station. One such system can be fluorescence combined with tagging as it provides significantly improved selectivity and sensitivity over some direct analysis techniques.

UV Spectroscopy

These systems are common tools associated with liquid chromatography devices. Again, this is a non-destructive technique that will provide unique identification capabilities across a broad spectrum of small molecule and biologic compounds. It is safe well-developed technology and is compact and requires little sample. This technique has a long history of utility there is a lot of data that can be used to help understand sample make up and compound identity. This system would need to be capable of being connected to the outflow of the Flow Chemical Reactor and for use with single samples via a cuvette sample holder. An autosampler would be nice but the failure of the last UV spectrometer was linked to its failed autosampler. Any automation will need to be thoroughly tested and both ORU will need to be considered as well as manual processes that can bypass automation when necessary.

Static, Dynamic, and Multiple Light Scattering

Static and dynamic light scattering are powerful techniques for characterizing soft matter and complex fluids for measurement of molecular weight, the mean square radius of gyration, the second virial coefficient, the diffusion coefficient, and the corresponding hydrodynamic radius. Additionally, in some cases, information on the shape and architecture of the molecules can also be gathered. These measurements are predicated on, among other requirements, single scattering events between incident photons and molecules or materials in solutions. A difficulty in measuring the local phenomena underlying the mechanistic characterization of soft matter and complex fluids including suspensions, foams, and emulsions, is that they tend to be opaque. Therefore, the scattering of light from the maze of liquid–liquid or gas–liquid interfaces causes incident photons undergo a random walk in the sample, and generally restricts optical imaging to the sample surface, to index matched emulsions, or extremely dry foams. However, multiple light scattering also opens up the possibility for new diagnostic tools. For example, the step-size in the random walk can give information about the size and packing fraction of the bubbles or droplets in the sample. Capabilities in both single and multiple light scattering would greatly enhance the structure and dynamics of soft materials and complex fluids in microgravity.

Shear Rheology

Rheology is the study of the deformation of a material under an applied field. While simple fluids display Newtonian behavior, i.e. the linear relationship with shear stress and strain rate, there’s a strong pure and applied interest in the flow and rheology of soft matter and complex fluids – particularly in the absence of a gravitational field. Rheological instruments typically control either the applied strain rate (both amplitude and frequency) or the applied stress (force per unit area) and measure the response of the fluid under study. Of particular experimental interest are shear flows and the importance of the no-slip boundary condition, dynamic studies of time-dependent rheological behavior, flow instabilities, multiphase systems, and electro/magnetorheological fluids. Because soft matter and complex fluids are often characterized exclusively in terms of their rheological behavior, experimental capabilities in rheological techniques – especially techniques that control the applied shear field, are important to include.

NMR

Nuclear magnetic resonance spectroscopy (NMR) measures the magnetic resonant frequency of the nuclei of individual atoms within a molecule. The NMR frequency of each nucleus is highly dependent on its electronic environment. An NMR spectrum provides detailed information about the structure of organic molecules that is not easily obtained by other techniques. NMR can also be used to assess purity of a reaction mixture. NMR is a non-destructive analytical method. ^{1}H-NMR is by far the most widely used method for characterizing organic molecules because it is fast and provides a great deal of structural information. So-called "benchtop" NMR spectrometers are uniquely suited for use on the ISSNL because they are small, require no cryogenic fluids or deuterated solvents, and they operate at low magnetic fields. Using Fourier-transform (FT) techniques built into the software, they can provide information that in the past only larger models could deliver. Most benchtop NMR spectrometers are easily incorporated into flow chemistry systems. Flow-NMR can be used to monitor reactions and characterize products.

Gas Chromatography (GC)

Listed last, this system is potentially the most basic and proven technology of the group of requested systems. It is a system for separating out different components of mixtures or reactions to get semi-quantitative measures of the make-up of the mixture. These systems have been a mainstay in most synthetic labs and the technology is robust. What makes them even more attractive is the ability to connect them directly to one of the analyzers already described above. GC Mas Spectroscopy as well as GC – IR systems are commercially available and safe to use. They require little material and maintenance is simple. They do require heating of the columns and there will be a need to adapt them to use on station. This system is destructive but would require little sample that could be shunted form the main stream without operator input. New technology "GC on a chip", a micro fabricated analytical system etched into glass, produces much less waste, takes up much less space, uses less power and would be easier to replace (less required maintenance). This can be considered as an alternative. Complexity and operational principle of detector technology may limit what is feasible to deploy on the ISS. TCD may be the most straightforward solution and require the least complex maintenance.

Additional Equipment

While the above-mentioned systems provide an overview of research facilities, some other facilities that also needs to be explored owing to additional capability improvement. One such system would be a short path length optical system (e.g., Nanodrop used for oligonucleotide concentration and spectrum measurement) owing to higher optical densities.

Overall Budget Required

The overall budget request for analytical tools is $9275000. This includes the actual units (two units one for implementation and one as a fully operational ground unit built to the same specifications for

FT-IR (2 units)	300000	
Implementation	600000	
Application ready	200000	1100000
Raman (2 units)	375000	
Implementation	600000	
Application ready	200000	1175000
Raman/Fluorescence	400000	
Implementation	700000	
Application ready	200000	1300000
UV (2 units)	250000	
Implementation	500000	
Application ready	200000	950000
Light Scattering (1 unit)	300000	
Implementation	500000	
Application ready	200000	1000000
Rheometer (1 unit)	150000	
Implementation	500000	
Application ready	200000	750000
NMR (2 units)	450000	
Implementation	800000	
Application ready	250000	1500000
GC (w/mass spec) (2 units)	350000	
Implementation	800000	
Application ready	350000	1500000
Total	9275000	

mirroring, controls and troubleshooting, their testing and conversion into flight ready units, installation and on-station testing and verification. This also includes training, operational software and sample preparation and testing materials.

The effort will also require launch, astronaut time and possible return services to deliver and set up the units and to return samples or entire units if need be. It is estimated that the costs associated with this part of the effort will be more than $20 million in costs associated with and in conjunction with normal ongoing operations that are already part of the ISS annual budget. There will also be other costs associated with incorporating these technologies into the ISS lab facility and developing the "Wet Lab". These other costs are associated with setting up the analytical lab facility (organizing power supply, positioning the hardware, sample preparation and associated materials, ongoing maintenance, and verification of system performance.) This is expected to cost between $1 and 5 million in the first year and between $500K and $1 million in subsequent years.

Conclusions

The above-mentioned facilities are crucial to provide wider access to soft matter research community and increase the overall impact of the soft matter research. We strongly believe that successful execution in this research campaign will improve the growth and impact of soft matter research in microgravity. The proposed facility will enable three key aspects of future research in space: Iterative and responsive science performed in a timely and more cost-effective manner, the ability to test soft materials in the microgravity environment, and future exploration efforts where sample return is not possible/practical. The ability to provide access to wider research community will also create a diverse and more inclusive soft matter research community.

References

[1] https://ntrs.nasa.gov/citations/20205010493