Picking, Sticking, and Clogging:
Characterization Approaches to Understand Particle Adhesion

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Project Overview

The adhesion of fine particles to metal processing equipment is a ubiquitous problem spanning industries from pharmaceuticals, food processing, construction, and agriculture.[1, 2] The high surface energy of most metal surfaces combined with the strong electrostatic attractive forces that act on small particles lead to strong adhesion. When these particles adhere so strongly, they can lead to damage to the processing equipment and defective parts.[3, 4] Clogging of extruders and hoppers can cause production delays and underfilling. Sticking and picking in the formation of compressed pharmaceutical tablets can lead to incorrectly sized medications and illegible markings, defects that currently account for significant waste in the industry.[5, 6]

Humidity further complicates the problem of unwanted particle adhesion. Capillary interactions can be much greater than the electrostatic adhesion forces, causing particles to adhere even more strongly to surfaces and to each other, exacerbating the clogging issues. The general consensus among particle adhesion studies indicates that maintaining a relative humidity below 50%RH will prevent meniscus formation, which is the driver of capillary adhesion. However, this number may vary depending on the hygroscopic nature of the particle and the hydrophobicity (measured by water contact angle) of the substrate. A deeper investigation into the role of humidity on particle adhesion is required to enable a complete picture of how to alter processing conditions to mitigate undesired particle adhesion.

There is a critical need to develop characterization tools that enable quantitative relationships between particle size, shape, type, substrate surface energy, surface roughness, temperature, and humidity. These characterization tools can then be used to evaluate the sticking potential between real world particles and substrates.[7] By coupling existing adhesion measurement tools in the Polymer Interfacial Mechanics Lab with new particle removal approaches, these characterization tools will fill the existing gap between theoretical predictions for particle adhesion with observations of real systems across industrial applications. **We propose to use flat air jets and contact mechanics tools to quantify the adhesion of particles to metal surfaces to achieve a quantitative measure of particle adhesion.**

Typically, particle adhesion has been considered at an individual particle level, utilizing an idealized model of a perfectly spherical particle on a smooth surface.[8-10] Many theoretical relationships have been developed that consider the electrostatic and capillary forces that lead to adhesion between rigid particles and substrates. However, tools to assess total adhesion of real-world particle/surface systems are still limited. The approach proposed here will emphasize scalable, applicable measurement tools development for a wide range of particles and substrates, moving beyond the single particle scale to investigate how particle ensembles and aggregates can be removed. We will use flat air-jet nozzles to apply shear forces parallel to the particle/substrate interface coupled with optical microscopy and automated image analysis tools to track particle removal with time and shear stresses. Leveraging existing tools and methods at the University of Delaware,[11-14] we will also incorporate substrate temperature, enclosure temperature, and relative humidity controls in the air jet particle adhesion test (AJ-PAT) set up. By constructing a robust testing platform that is substrate and particle agnostic, the efforts of this project will produce a characterization tool that can be generalized to the diverse array of materials systems and production environments of the IFPRI Member Companies.

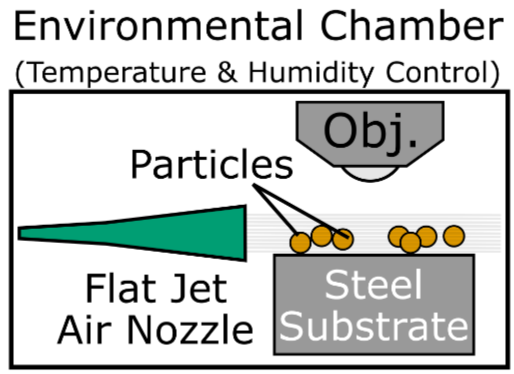


Figure 1: Air Jet Particle Adhesion

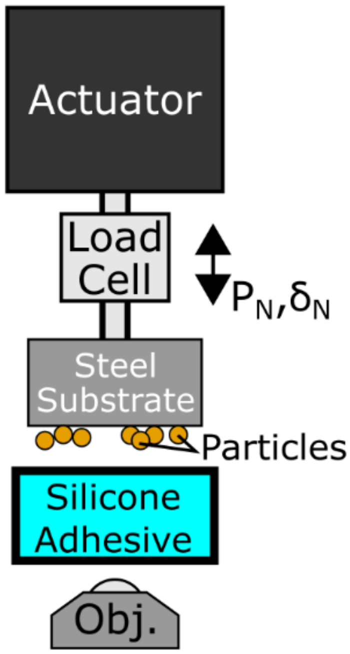


Figure 2: Contact Adhesion Tester for Particle Removal

Following development of the AJ-PAT system, we will further screen and evaluate material formulations as a function of interface composition and particle/surface application history through contact mechanics adhesion tests. These parallel experiments will be compared with the AJ-PAT results to verify the results of the air jet test method. We will utilize existing custom-built *in situ* mechanical contact adhesion testing (CAT)[11, 13, 14] coupled with high resolution imaging to investigate how particles detach from the various substrates.

Our objective is to develop a testing platform with robust mechanical and environmental controls and to demonstrate a scalable process for evaluating particle adhesion.

Value to IFPRI Member Companies

The proposed research activities (discussed in detail below) describe a novel investigation of particle adhesion through the development of a new test method and the subsequent characterization of particle removal as a function of materials, environmental, and mechanical factors. We additionally propose to measure a range of particle/substrate systems recommended to us by the IFPRI Member Companies. Expected results will advance fundamental scientific knowledge within the particle adhesion field, contributing to antifouling approaches and particle removal methods for multiple particle/substrate systems. This project will also develop a new particle adhesion measurement tool that can be employed to evaluate new particle/substrate adhesion effects as they are being developed and implemented within the IFPRI Member Companies.

The **value of the proposed research to the IFPRI** will be the characterization of particle adhesion as a function of surface preparation and environmental factors. Our fundamental studies of particle removal and parametric studies on the adhesion of several common particle/substrate materials systems will be used to develop models relating the particle removal efficiency to particle size, shape, surface energy, and substrate surface type and roughness. The motivations and long-term goals of this research are highly interdisciplinary and of immediate interest to the fine particle manufacturing community. The Ph.D. student researchers involved in this work will develop experimental research and technical communication skills that will prepare them for future careers in a range of particle manufacturing fields.

Polymer Interfacial Mechanics Lab Facilities and Track Record

PI Davis's Polymer Interfacial Mechanics (PIM) Lab (University of Delaware, Spencer Engineering Laboratory labs 007, 317, and 318) is comprised of three rooms for a total of 1250sq. ft. of lab space dedicated to instrument fabrication, micromechanical testing, and optical microscopy. The PIM Lab microscopy space has been modified to be a "quasi-cleanroom" (door curtains, threshold tape mats, HEPA filtration, and dark room curtain) to prevent environmental contamination that can be detrimental to microscale mechanical and optical characterization experiments. The basement lab space is ideal for optical microscopy work, with adjustable intensity overhead lighting, no windows, and sub-grade elevation to reduce external vibrations. The sample preparation labs are equipped with all of the standard equipment required for routine particle processing and handling. An environmental control HVAC system has been installed in SPL 318 to maintain temperature and humidity conditions independent of the building's air handling system. All tools required to design, manufacture, and program the AJ-PAT are readily available in the PIM lab space and the recently renovated Spencer Lab Design Studio, comprised of 13,000sq. ft. of machining tools.

The expertise of the PIM lab lies in our approach of designing and building novel characterization tools on a small scale coupled with simultaneous optical visualization. As a recently inducted Robert L. Patrick Fellow of the Adhesion Society, PI Davis's career has spanned nearly two decades of fundamental adhesion engineering research[14-17] and micromechanical characterization tools development.[12, 13, 16, 18, 19] She has applied these approaches to adhesion questions in many industrially-relevant materials systems including rubber layers in tires,[16] pavement marking materials for road construction,[20-23] 3D printed layer cohesion in polymer additive manufacturing,[11, 24, 25] medical bandage peel adhesion,[26] and polymer matrix composites.[27] Her specific experience in particle adhesion is evident through several nanocomposites projects, focusing specifically on nano and microcellulose crystals and fibrils.[19, 28-30] PI Davis worked on particle powder adhesion to tablet punches (Purdue University Senior Design 2018) and is currently focused on particle surface fouling of silicones. The PIM lab is a space dedicated to training the next generation of adhesion scientists and engineers with skills in the fundamentals of surface science and the practical understanding of how these fundamentals can be applied in the real world.

Objectives and Expected Outcomes

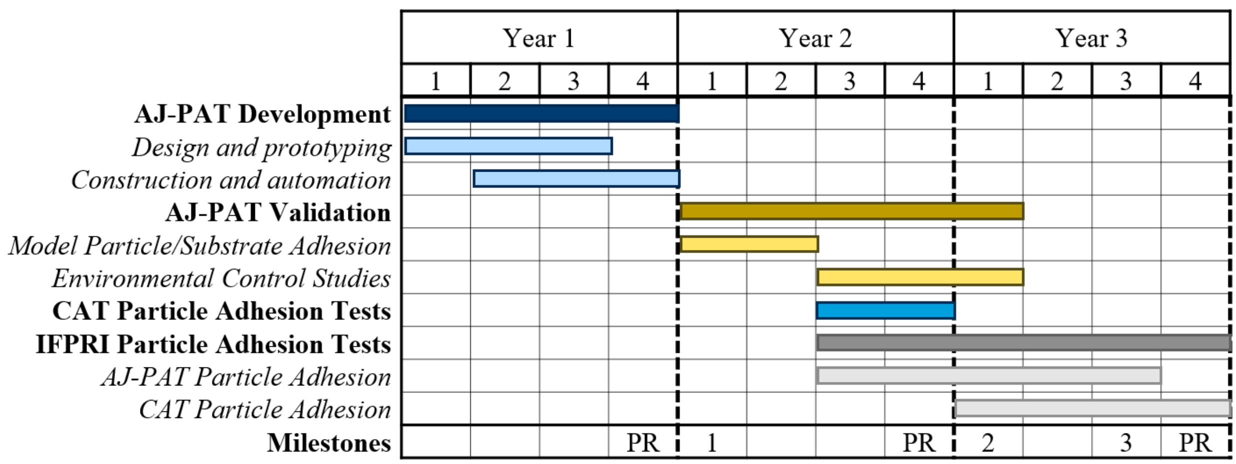
Specific outcomes of the work include establishing a quantitative understanding of how properties such as particle material and size, substrate surface energy and roughness in addition to environmental conditions (i.e. temperature, humidity) affect the adhesion and removal of particles. The project will be broken down into several key objectives.

1. Design, assembly, and automation of AJ-PAT
2. Measurement of particle removal efficiency with AJ-PAT in model particle/substrate system
3. Validate measurements of particle removal efficiency with CAT in same model particle/substrate system
4. Measurement of IFPRI member-determined particle/substrate systems with both testers

Upon completion of this project we will know how the choice of particle (based on size, shape, modulus, and surface energy), substrate (based on surface energy and roughness), and environment (based on temperature and humidity) can influence particle removal. Given the vast parameter space possible with this number of variables, we will rely on input from the IFPRI members to focus our efforts on key parameters and materials as the project progresses. These measured particle adhesion relationships will be combined to form a semi-empirical model which can be used to better design particle processing conditions and handling equipment.

The research will be conducted by one Ph.D. student, advised by the PI Davis. Funds are requested to support 50% of one student's time over three years. All materials and experimental equipment are already available in the PIs' Labs and will be utilized for the proposed experiments.

Proposed Timeline



Milestones: (1) publication on AJ-PAT device (*Rev. Sci. Instruments*), (2) publication on model particle adhesion and CAT calibration (*ACS Applied Polymer Materials*), (3) publication on industrially-relevant particle adhesion (*Part. Sci. & Tech.*), (PR) progress report to IFPRI

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