Machine learning tools can help accelerate materials discovery and design, but a large data set is generally needed to train the models. Therefore, expanding the use of machine learning to new research problems goes hand in hand with the need for data generation. The ability to predict mechanical properties of soft materials, such as adhesion, could benefit from larger data sets. Here, Chen et al. detail a high-throughput experimental methodology for testing soft adhesives that is capable of testing on the order of $10^3$ samples per run and provides a means of producing the data sets required for machine learning.\(^1\)

Methods for the acquisition of large data sets for mechanical testing are limited. A high-throughput methodology for adhesives using centrifugation could help bring big data to the field of adhesion.

The ability to predict mechanical properties of soft materials, such as adhesion, could benefit from larger data sets. Here, Chen et al. detail a high-throughput experimental methodology for testing soft adhesives that is capable of testing on the order of $10^3$ samples per run and provides a means of producing the data sets required for machine learning.\(^1\)

Figure 1. (a) Experimental methodology for centrifugal adhesion testing. (b) (top) Before and during centrifugal adhesion testing, the top well has strong adhesion; the bottom well has weak adhesion, and particles detach. (bottom) Probe tack experiment where the probe is brought into contact with adhesive (left) and subsequently retracted (right). (c) Output of the centrifugal adhesion test; each column has a progressively larger fraction of adhesive, and each row shows the number of particles that remain after being subjected to a specified force. Panels a and c are reproduced with permission from ref 1. Copyright 2021 The Authors. Published by American Chemical Society.

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inherent interplay between surface interactions and bulk rheology.\(^2,3\) In particular, the relationship between formulation and adhesive strength can rarely be predicted \textit{a priori}. Adhesives are also increasingly becoming multifunctional, for example, enabling monitoring of body functions and meeting the need to operate under extreme conditions (such as in contact with sweat, water, or at high temperature).\(^4\) Therefore, the ability
to dial in the chemistry that would yield a predetermined adhesive performance through machine learning could enable the development of better adhesives.

While high-throughput adhesion testing methods exist, they can be challenging. For example, a method for axisymmetric adhesion measurements with simultaneous contact imaging requires careful alignment of all lenses and cannot provide measurements for each individual lens’ displacement. Other methods require custom machined instruments, or instrumentation than can be cost prohibitive for many. In their work, Chen and co-workers report an adapted version of a standard adhesion test using a common laboratory centrifuge. To put their work in context, the characterization of adhesive strength is typically performed one sample at a time. A common adhesion test is the probe tack experiment, which consists of bringing a probe into contact with the adhesive film (substrate), followed by its retraction while measuring the force. In addition to obtaining adhesive strength, probe tack experiments allow for the control of strain rate and often imaging of the contact region. In the method of Chen et al., they first cast PSA films with uniform thickness in a 384-well plate; the PSA in each well acted as independent substrates. Colored particles are then deposited on the film and act as probes. To detach the particles from the PSAs, the 384-well plate is placed “upside down” (with particles facing out) in the centrifuge and spun at a given rate (rotations per minute). The adhesive strength is then inferred from a visual inspection of the fraction of particles detached after centrifugation.

Centrifugation applies an outward force determined by the mass of the species, angular velocity, and distance from the axis of rotation. Prior centrifugation force measurement (CFM) techniques demonstrated the ability to obtain a large number of data points for a single sample or condition, i.e., parallel data acquisition. However, the capability of CFM to test adhesion for multiple conditions more efficiently than a standalone single sample instrument had yet to be demonstrated. The use of a multiwell plate allows for up to 384 unique formulations or conditions to be tested simultaneously, up to 1536 if you use each position in a 4-place rotor. Upon removal of the plate, the number of spheres stuck to a film can easily be quantified by color intensity. The more spheres that have detached, the less intense the color, thus yielding a simple assay for determining the force range required for particles to transition from predominantly attached to predominantly detached. To validate their results, the authors showed agreement between the centrifugal adhesion test and probe tack experiments using the same materials (however, a correction factor of 1.6 is needed). CFM is not yet a replacement for probe tack experiments. For example, control of strain rate, stress—strain curves, and mode of failure are important characteristics that cannot currently be resolved with CFM. Instead, the method can serve as an excellent first pass filter to screen candidates worth investigating further with more time-consuming techniques.

The framework of the methodology reported by Chen et al. could be modified for more widespread applications in adhesion science. For example, a common option for centrifuges is temperature control, typically from −10 to 40 °C. This option would enable temperature control for experiments and span temperature ranges useful for studying ice adhesion to adhesion in physiological environments. Additionally, with slight modification, the centrifugal adhesion test could be expanded to underwater adhesion or experiments with extended contact times. Finally, with a bit more modification, a camera could potentially be incorporated to capture debonding behaviors and identify the strain rate or mode of failure. Ultimately, a true test for the method will be its future implementation with machine learning tools to develop novel high-performing formulations for adhesives.

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