

## **Interfacial Engineering**

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**Lecture-37**

**Force-measuring techniques: Direct and indirect measurements**

**Indirect and direct force methods; peeling and adhesion force; surface force apparatus (SFA); atomic force microscope (AFM)**

Welcome back to this video lecture. We will be looking at the force measuring techniques. Okay, some of these techniques are labeled under direct and indirect methods. Okay, we have looked at the various types of forces, right? Van der Waals forces of attraction and electrical double layer interaction, right? But we have used some, you know, fundamental equations. Then we try to derive and develop the equation for interaction between two particles, to obtain pairwise interaction between a planar surface or between two particles, right? But in this video, we will look at some of the measurements we can perform to assess the forces, right?

To understand or to understand the interaction, you know, between the surfaces, or to determine the forces directly or indirectly, right? That is precisely what we are going to look at in this video. Let's begin. Yes.

Time: 1.41 mins

## INTRODUCTION



<b>Definition</b>	<u>Forces govern stability, aggregation, and interactions in colloidal systems.</u>
<b>Significance</b>	<u>Crucial in studying adhesion, wetting, emulsion stability, and nanoparticle interactions</u> ✓
<b>Classification</b>	<u>Direct - Measures interparticle forces explicitly</u> <u>Indirect – Estimates based on Secondary effects like deformation or energy dissipation</u>

Indirect methods are those in which we use some macroscopic property or phenomenon to deduce information about (colloidal or intermolecular) interaction forces

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So why? First, let us ask a question. Why do we need to measure forces? Only then will you be able to say whether a given dispersion is stable or whether the given system is stable. For example, suppose you want dispersion to be stable. In that case, you want to promote electrical double layer interaction between the particles, mainly when dealing with the liquid-solid phenomena, to prevent aggregation, right? So, for that, one has to understand the type of forces involved in the systems.

And if you want to understand or study the interaction in colloidal systems, you must understand and address the forces involved. So, we need to examine various direct and indirect methods to understand the types of forces involved. The significance part, if you look at it, is very crucial in studying the adhesion phenomena, wetting, emulsion stability, and nanoparticle interactions. So in these cases, you need to understand the forces and the various types of forces involved. And for that, we need to determine the forces. Different methods can be classified into two.

One is the direct method. So this uses some probe. It is, you know, an in situ method. So it uses some probe to measure the, you know, the forces between the, surface as a function of you know several factors one of the factors could be the distance between the probe and the surfaces okay the other one will be indirect method so you will have you know several methods you know labeled under this category So you will be measuring some property, okay, some macroscopy property, right, to deduce information about the macroscopy property like intermolecular you know interaction forces or colloidal interaction forces right so that is nothing but indirect methods okay that is precisely what we are going to look at under indirect methods.

Time: 4.26mins

### Indirect Force Methods

Microbalance or indentation tests are used to deduce adhesion force

$$W_{12} = \gamma_1 + \gamma_2 - \gamma_{12}$$

Peeling force

Adhesion force	Peeling force
Force that holds two surfaces together - vdW, ES, covalent, H, and ionic	Force required to detach an adhered material via peeling
Surface chemistry, roughness, electrostatic interactions are the influencing factors	Peeling angle, material properties, adhesion energy
Applications: Nanoparticle adhesion, coatings, biomedical interfaces	Adhesive tapes, protective films, wound dressings

Hiemenz, P. C., & Rajagopalan, R. (2016). *Principles of Colloid and Surface Chemistry, revised and expanded.*

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So let's start with the indirect force methods. The first set of methods that we will look at is the adhesion force and peeling force.

Remember, we have looked at the adhesion force, work of adhesion and work of cohesion during the module two, where we, you know, shown that by computing the surface energy of block A, and surface energy of block B and subtracting them with the interfacial energy of block A and B you will be able to compute the work of adhesion which is expressed in the form of you know energy per unit area joule per meter square okay so we have looked at this during the module two now exactly what we are doing is by knowing the, you know, force required to, detach them by detach, force needed to detach the particle, I mean, these two surfaces, okay, away from each other by, you know, computing the, you know, that particular force, one can calculate what will be the adhesion force, right? And then one can, you know, um, get more insight into you know type of forces whether it is due to you know hydrogen bonding ionic bonding and van der waals interaction so those aspects can be studied later okay and what governs this force is the surface chemistry between the surfaced roughness and electrostatic interactions at the boundary between two surfaces. In the case of peeling force, you will be computing or deducing the force, adhesion force, or peeling force by calculating the force required to detach an added material via peeling. So the governing factor here will be the certainly peeling angle, right? So that will affect the peeling force. And material properties, right? And adhesion strength. So these are governing factors when it comes to peeling force. And these are some applications where you will encounter the, you know, you will, you know, use this adhesion force quite often. That is the nanoparticle adhesion coatings, right? Whenever you deal with coatings, you

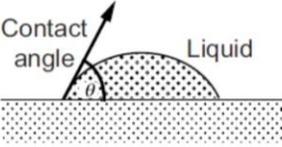
want to know the adhesion strength of the coating material.

Over the surface and biomedical interfaces. So these are some applications you will often deal with regarding adhesion force. So, some situations where the peeling force will be required to calculate are the adhesive tapes. When you design adhesive tape, right? for specific application you will need you will have to calculate this peeling force and protective films when you want to develop the protective film okay to prevent the surfaces from rusting you know so from for those aspects and wound dressings so here also you will be you will be required to calculate what is the peeling force, right? So these are some applications when it comes to peeling force. Next, these are indirect methods again, okay? By calculating these forces, you will understand or assess the forces between the surfaces. Or liquid between the, you know, surfaces, mainly when you deal with the adhesion force as well as the peeling force, okay?

Time: 8.58 mins

**Contact angle**





$$W_A = \gamma_{LG}(1 + \cos \theta)$$

Contact angle measurements	
<b>Overview</b>	✓ $\theta$ helps to determine adhesion forces, surface tension, and interfacial energy
<b>Challenges</b>	✓ Surface roughness and heterogeneity affect accuracy. ✓ Contaminants and humidity can alter results.
<b>Applications</b>	✓ Colloid Stability: Wettability influences aggregation and dispersion. ✓ Bioadhesion: Protein adsorption, cell adhesion, and biocompatibility. ✓ Emulsions and Foams: Wettability determines droplet stability in emulsions. ✓ Microfluidics and Lab-on-a-Chip Devices: Surface wetting is critical.

11:55 / 27:14 | [Citation: P. G., & Rajagopalan, R. (2016). Principles of Colloid and Surface Chemistry, revised and expanded]

Next comes the contact angle, okay? Again, based on the Young-Duprey equation, you will be familiar with because we have developed this equation during our module two.

So, by knowing the theta, you know, of the liquid, okay, contact angle established by the liquid with the surface, you can calculate the adhesion strength or adhesion force, I mean adhesion energy in this case. Okay, for that, you need to know the gamma component of the liquid you will be dealing with, and this is very easy to obtain, right? In contrast, the theta one has to be one has to determine okay using sessile drop method okay so that you will know theta as well as gamma lg by understanding these two component you can

calculate the adhesion energy right in this case work of adhesion right yeah So this particular aspect, this contact angle will help you determine the adhesion force, not only adhesion force.

Sometimes, if you know the work of adhesion, you can even calculate the surface tension and the interfacial energy, right? So, the challenges involved in this approach are that this method cannot be applied if the surface is rough and heterogeneous, because it affects the accuracy. You will be discouraged from using this method if the surface is exposed to contaminants and humidity. Because these factor also certainly affect the result and when it comes to application you will be able to use this method to you know address the wettability okay of when it comes to colloidal stability And bioadhesion, so when you want to calculate protein adsorption, cell adhesion and biocompatibility, this approach will be undoubtedly helpful. Again, in emulsions and foams, wettability determines the droplet stability.

And last but not least will be the microfluidic and lab-on-a-chip device. Surface wetting is a crucial factor in this application. So, one must know the wetting behavior of a liquid spread on a surface. So if you want to understand the adhesion between the film thickness the resistive film thickness resistive film and the surface you need to know the wetting behavior so so here it is undoubtedly going to be very useful right so this is again indirect method okay.

Time: 12.08mins

### Soap and liquid film

- Polymer films in SC devices
- Lubrication layers
- Foam films

Capillary pressure, disjoining pressure, adhesion forces, and molecular interactions

$$\Delta P = \frac{2\gamma}{R}$$

$$\Pi_{VDW}(h) = \frac{-A}{6\pi h^3}$$

$$\Pi_{EDL}(h) = \Pi_0 e^{-h/\lambda_D}$$

Method	Principle	Measurement Range	Applications
Interferometry (White Light or Monochromatic)	Thin-film interference patterns correlate with film thickness	1 nm – 10 μm	Soap films, oil films, emulsion layers
Ellipsometry	Measures change in polarization upon reflection	0.1 nm – 500 nm	Adsorbed liquid films, surfactant layers
X-ray Reflectivity (XRR)	Measures reflected X-ray intensity to determine film structure	1 Å – 100 nm	Nano meter scale liquid films, polymer coating
Spectroscopic Reflectometry	Analyses reflected light spectra to determine film thickness	1 nm – 10 μm	Surface coating surfactant layers

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Soap and liquid film so here what we will be doing is we will be using several spectroscopic technique okay to determine the film thickness okay it could be due to soap film or it could be liquid film right confined between you know surfaces right to surfaces whereas it could be due to soap film formed by some surfactant right in water so if you want to understand this so if you're going to understand the force of interaction between the you know soap film and the surface or liquid film and the surface you can perform several of these techniques to understand you know what kind of forces involved right so so this interferometry if you use interferometry so basically you will be assessing, right, finally, you will be able to determine or calculate the capillary pressure, disjoining pressure, adhesion force, and finally, you will be able to assess molecular interactions, right? So, for that, what we need to do first is understand the film thickness. Once you know that, you can plug these values into the respective equation and calculate the capillary or disjoining pressure, Van der Waals and Van der Waals forces of attraction, or electrical double layer interaction by knowing the film thickness.

So, how we measure the film thickness is essential. Each method offers a different strategy through which one can use the film thickness, which is measuring the film thickness. Still, each method has limitations as in it is these methods are applicable only in this measurement range so depending on the measurement range one has to pick up the suitable method so in the case of interferometry based on white light or monochromatic light So basically what you will be doing is you will be, you know, performing the, you know, thin film. Sorry, you will be measuring the film thickness by generating the interference pattern. So, based on this interference pattern, you can determine the film thickness using white or monochromatic light. Once you know the film thickness, you can correlate that to find out the capillary pressure or the disjoining pressure okay and later you can assess you know the type of forces involved you can infer about Van der Waals forces or the electrical double layer you know interaction and all that right right that is all about the interferometry okay this is applicable in the range between 1 nanometer to 10 micron meter in the case of ellipsometry what we do is we measure the change in the polarized light okay you know of reflected light okay so basically it records the amplitude change and the phase change of the polarized light okay based on which you can get several information like film thickness and, you know, the refractive index and surface interaction. So, based on which you can, you know, address or determine film thickness, refractive index, and surface interaction. So, by knowing this information, you can relate this to, you know, inferred you know the type of forces in that you want to compute okay then comes the x-ray reflectivity okay so so x-ray reflectivity the reflected x-ray intensity obeys the Fresnel law okay.

So, the Fresnel law describes how light can reflect or transmit when it passes through an interface with two optical media, right? OK, especially when light passes through a liquid-solid or liquid-gas interface system.

OK, when it passes through one bulk phase to another bulk phase, it encounters different optical properties. Right. So, because of this, it generates a different interference pattern. By using such an interference pattern, one can deduce the information about the thickness of the film and electron density, right? And molecules' interface roughness and layering are in confined films, right? Based on which you can measure, capillary or disjoining pressure, right? And you can, you know, talk about whether the type of force involved or, you know, Van der Waals force or electrical double layer interactions by, you know, deducing this information, you can comment on whether the forces of interactions are due to Van der Waals or electrical double layer, okay? So that's how the force measurements are done indirectly through these spectroscopic techniques, okay? Here comes the direct force methods, nothing but surface force apparatus.

We have looked at indirect methods, and now we will look at some direct force methods.

Yeah, even before this, I forgot to mention the additional technique under the indirect method. So that is nothing but a coagulation study.

Time : 18.45 mins

### Soap and liquid film

- Polymer films in SC devices
- Lubrication layers
- Foam films

Capillary pressure, disjoining pressure, adhesion forces, and molecular interactions

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21:15 / 27:14 Benz, P. C., & Rajagopalan, R. (2016). Principles of Colloid and Surface Chemistry, revised and expanded

So, by performing the coagulation study, one can even assess the Van der Waals forces of attraction.

So this method of assessing the Van der Waals forces of attraction is straightforward. So, what one can do is, let's say, you systematically conduct the coagulation study by adding the salt at a regular interval.

So by knowing the critical concentration at which the surfaces or the colloidal particles tend to aggregate, you know, by determining the critical salt concentration, we can compute, you know, what is known as the electrostatic force. What we can do is, let's say, we know that the coagulation occurs when these particles or surfaces tend to overcome the electrostatic repulsive force between them. So by knowing this concept, what we can do is, since we identified the critical salt concentration, at that particular concentration, one can measure the zeta potential value. The other parameter is called the Debye-Huckel parameter

K.

So by measuring these two parameters at the you know a critical salt concentration we can substitute these values you know back into the equation that we derived in the previous lecture that is nothing but you know electrostatic force due to electrical double layer interaction there if you plug in these values what we will get is the electrostatic repulsive force itself okay since we know the electrostatic repulsive force. We also know that coagulation occur when the surfaces you know overcome the electrostatic repulsive force right so we know that at this particular critical salt concentration these particles or surfaces tend to coagulate which means that they have overcome the electrostatic repulsive force So, by computing the electrostatic repulsive force indirectly we can calculate or compute the Van der Waals forces of attraction. So, in this way, you can indirectly assess the Van der Waals forces of attraction. So, one has to systematically conduct this coagulation study to understand the strength of the Van der Waals forces of attraction between the surfaces or the colloidal particles.

Time : 21.35mins

## Direct force methods



### What is SFA?

- The Surface Force Apparatus (SFA) is used to measure normal and shear forces and study interactions between two surfaces at nanometre resolution, i.e., 0.1 to 10 nm.
- It primarily measures van der Waals, electrostatic, and steric forces.

### Principle of Operation

- Two atomically smooth, transparent surfaces (typically mica) are brought into controlled contact.
- White light multiple beam interferometry is used to measure the separation distances.
- Forces between the surfaces are determined by deflection of a spring system.
- Piezoelectric actuator allows to systematically vary the separation distance (F vs d).

### Key Features

- Measures forces in the nanoNewton (nN) to microNewton ( $\mu\text{N}$ ) range.
- Capable of detecting surface interactions in liquids, vapors, and air.
- Force vs. distance information is useful for colloidal interactions, adhesion, lubrication, and molecular forces.



21:44 / 27:14

Next, we'll move on to the direct force methods. There are a couple of methods.

So, you know, one, I mean, one of them, so the first and foremost technique we can see is the surface force apparatus, okay, SFA, is often denoted as SFA. So this measures regular and shear forces due to the interaction between two surfaces. Right at nanometer resolution, usually between 1 and 10 nanometers, a few tenths of nanometers, an okay separation distance. So, by calculating and measuring these forces, one can obtain information about the difference between force and distance, that is, the separation distance. The principle of operation is straightforward. So one has to take the mica surfaces, right? Why is mica preferred? Because when the surface is cleaved, it becomes very flat, and the smoothness is comparable to the atomic scale.

That's why people often prefer these mica surfaces. When you bring these mica surfaces together, right means that one of the surfaces will be rigid, and the other will be moving. The moving surface will be attached to the spring system. So when these surfaces are brought together in a very close proximity, that is at a separation distance of, let's say, a few nanometers, there will be molecular forces or atomic forces that will cause the deflection in the spring system. By determining this deflection, one can calculate information about the force.

The separation distance can be measured using the interferometric technique. One can measure the separation distance by using the white light interferometric technique. By knowing the separation distance and the force, it is easy for us to calculate, you know, to understand the type of forces involved and the magnitude of the forces, right? So, an important point to note is that the piezoelectric actuator is often used to systematically vary the separation distance between the surfaces. So some of the key features of this technique are that you can measure the force in the range between at least micronewton and nanonewton. You know range right the force versus distance information is beneficial for colloidal interaction for understanding the colloidal interactions okay and to assess the strength of adhesion in lubricants lubrication and to understand more about the molecular forces okay next comes in this in a category next comes the AFM which is nothing but the high resolution technique okay so what it does is it measures the surface topography and mechanical property by deducting the forces between a sharp tip and the sample so basically we like previous one we you know, we detect, we, I mean, capture the force between the, we obtain the information about the forces between a sharp tip and a sample, okay? Right, so here, like SFA, here, the sharp tip is mounted on a flexible cantilever, because when the sharp tip and the sample interact at a very close distance with the separation distance of let's say a few nanometers in distance, the atomic forces of the molecular forces will cause the cantilever to deflect.

As a result of interaction between the surfaces, these forces will cause the will make the cantilever to deflect okay by determining this deflection one can again collect the information about the force versus the separation distance okay some of the key features of this technique is you can measure forces in the range between nano newton to micro newton okay like your SFA, right? Like here, the SFA can also obtain what we have seen in a similar range. So this technique can be operated in contact mode, tapping mode, and non-contact mode. Remember, we have looked at the methods and a few characterization techniques. We have given enough insight into some characterization techniques in module one. If you can go back and refer to lecture number three, you will be able to get more insight into these techniques. OK, so we will stop here and continue from the following lecture. Thank you.