

# **Novel Cellulose Nanoparticles for Potential Pharmaceutical & Personal Care Applications**

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IPR 2010



## **OUTLINE**

### **POLYAMPHOLYTE MICROGELS**

#### **INTRODUCTION**

#### **CHARACTERIZATION RESULTS**


### **NANO CRYSTALLINE CELLULOSE**

#### **INTRODUCTION**

#### **BINDING INTERACTION STUDIES**

#### **FUTURE WORK**

#### **SUMMARY**



**CHITOSAN - CARBOXYMETHYL  
CELLULOSE POLYELECTROLYTE  
MICROGELS**

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# POLYAMPHOLYTE MICROGELS

## POLYAMPHOLYTES

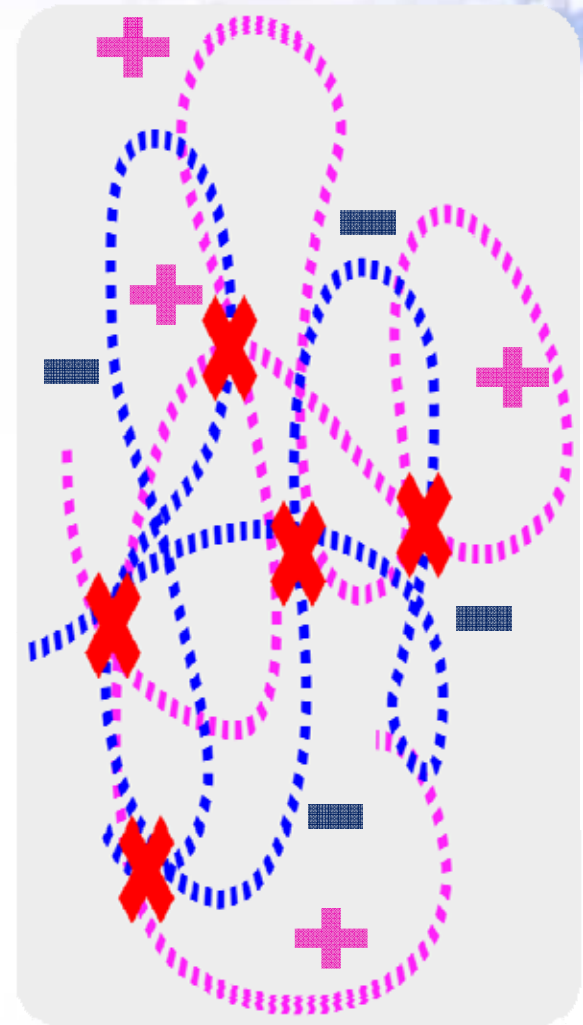
Polymers consisting of positive and negative charges

## MICROGELS

Crosslinked polymer particles

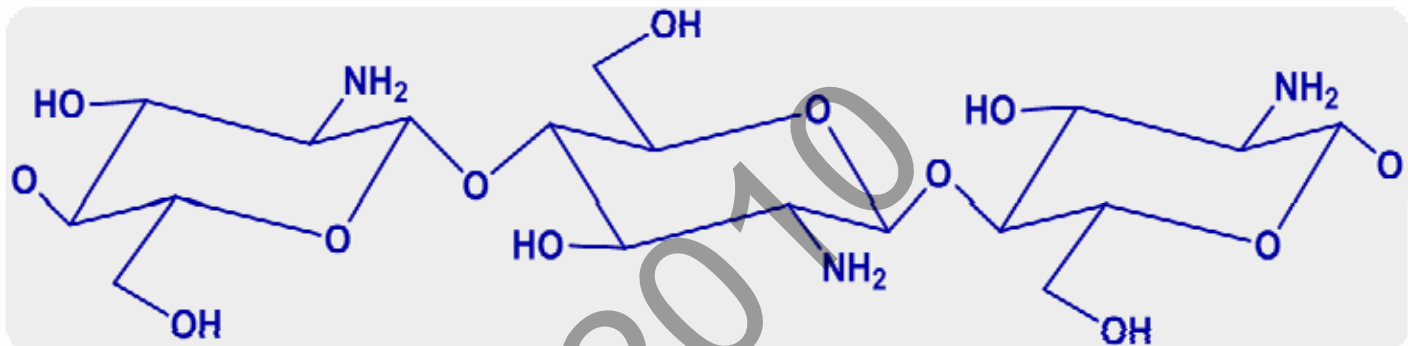
## POLYAMPHOLYTE MICROGELS

Crosslinked particles capable of possessing positive and negative charges

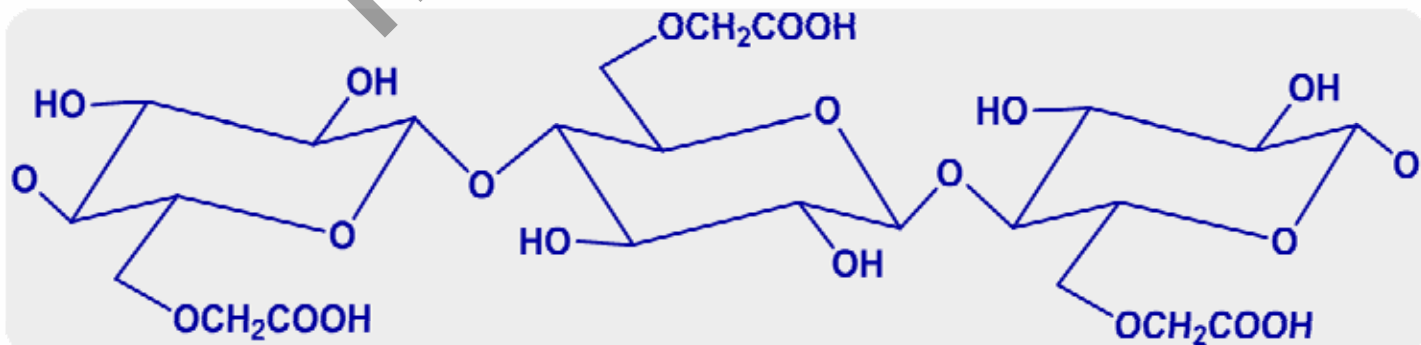


# PROPOSED SYTEM

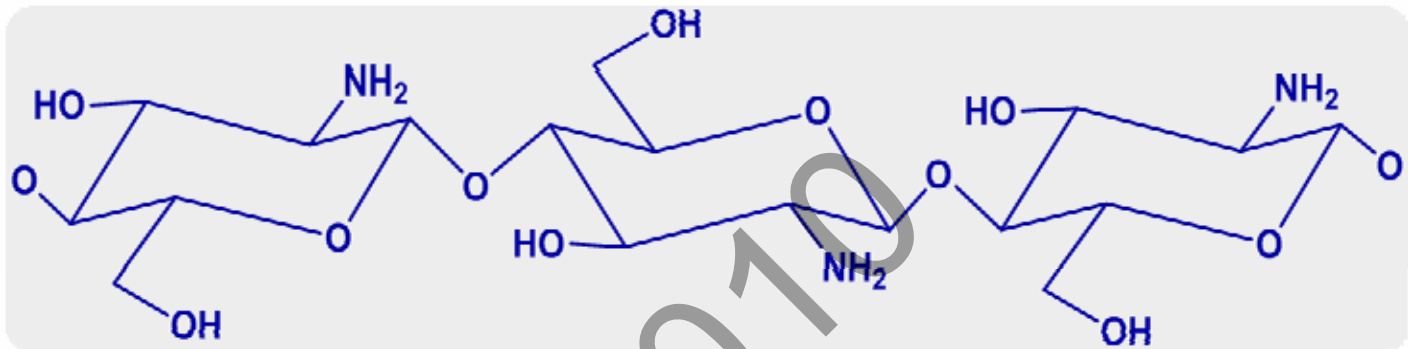
## CHITOSAN



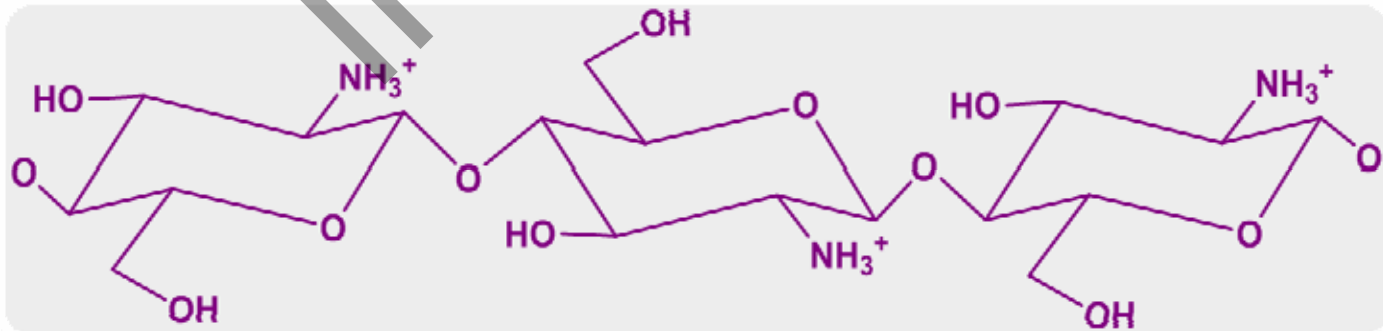
## CARBOXYMETHYL CELLULOSE



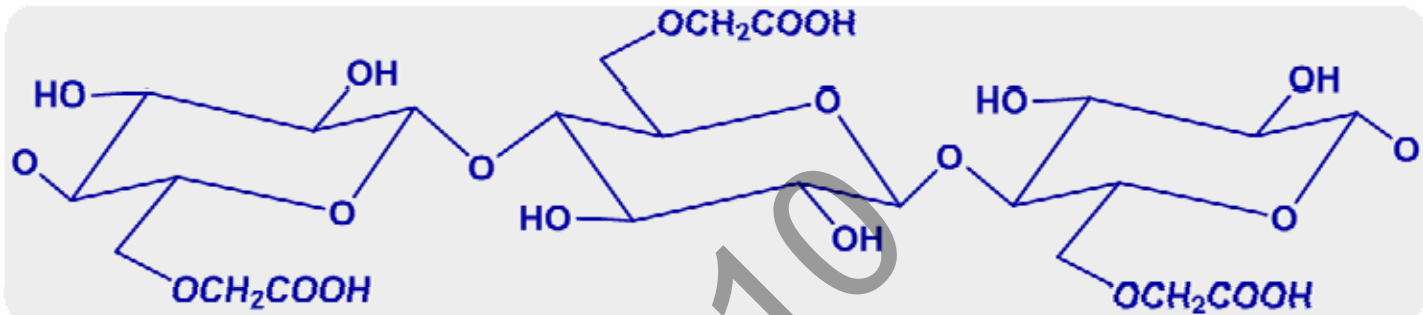
## CHITOSAN AT LOW pH



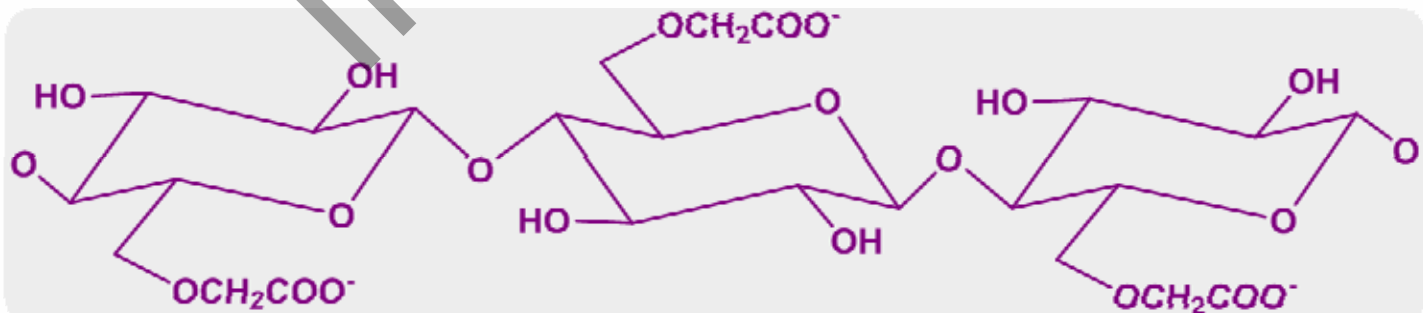
$\text{H}^+$



## CARBOXYMETHYL CELLULOSE AT HIGH pH

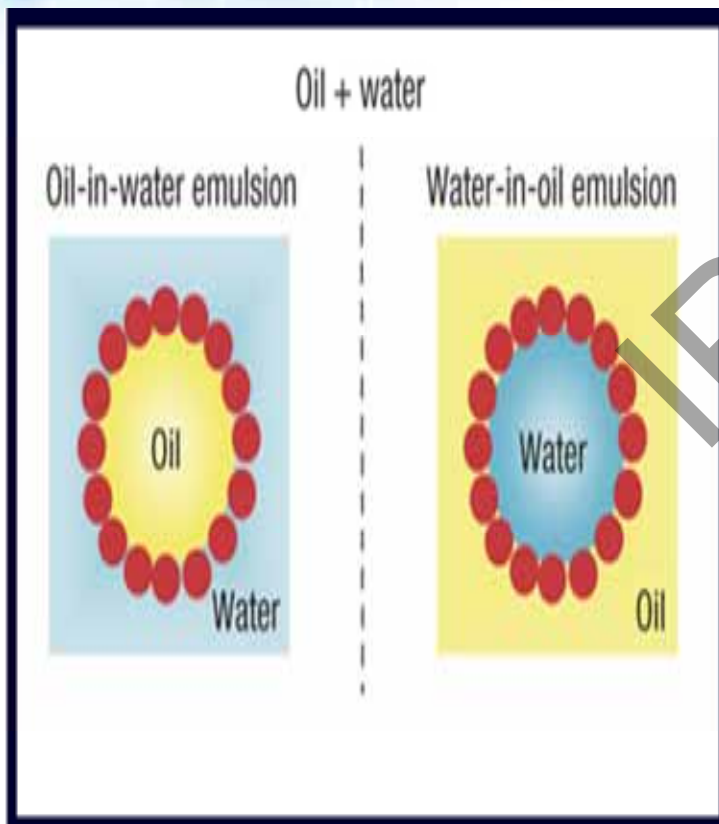


$\text{OH}^-$



## SYNTHESIS TECHNIQUE

### INVERSE MICROEMULSION POLYMERIZATION



1. Thermodynamically stable microemulsions

2. Non ionic surfactants

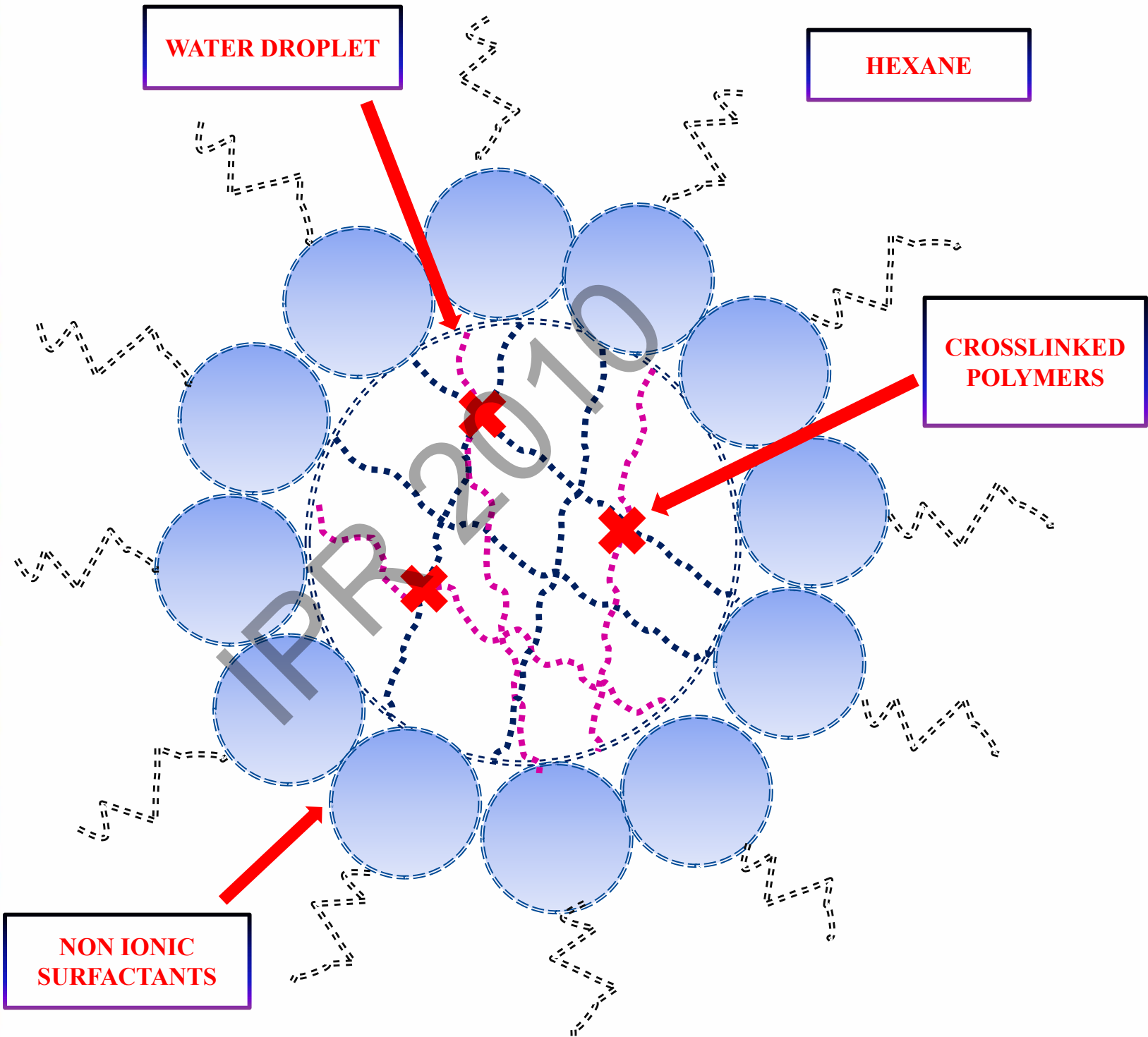
3. W/O emulsions at high temperatures

4. Water droplets act as nano - reactors

5. Polymerization & Crosslinking in the water droplets

6. Beneficial for water soluble polymers

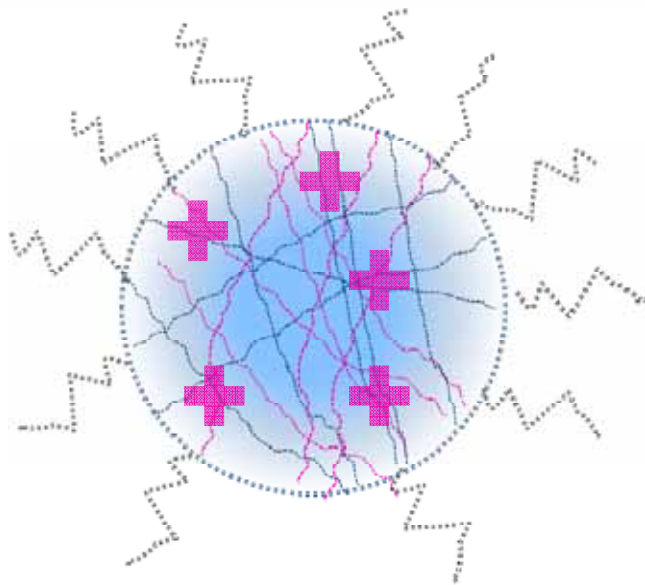




## pH RESPONSIVE BEHAVIOR OF POLYAMPHOLYTE MICROGELS

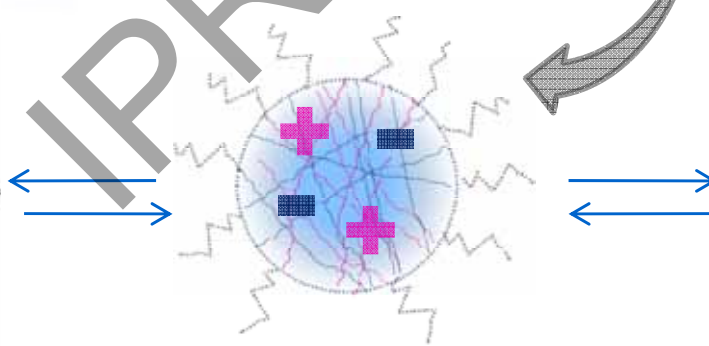
Steric stabilization is important !

Microgel Swelling



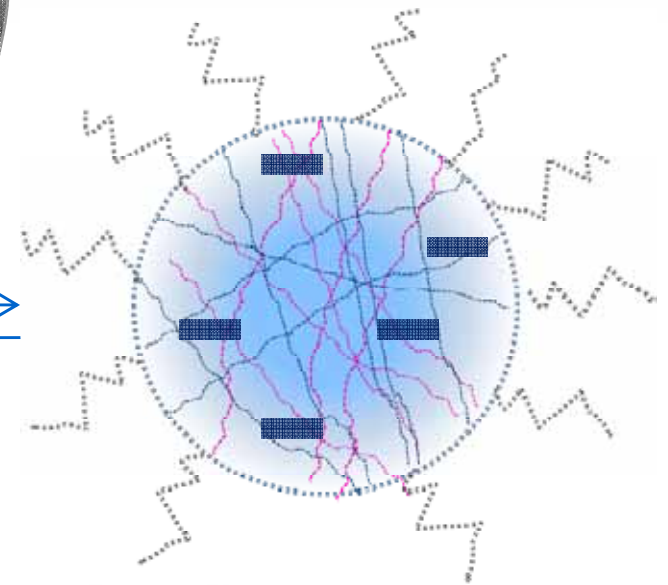
Low pH

Microgel Deswelling



Neutral pH

Microgel Swelling



High pH



**ADVANTAGES**

**BIOCOMPATIBLE**

**BIODEGRADABLE**

**TUNEABLE SIZE**

**NON TOXIC**

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# CHARACTERIZATION RESULTS

**Potentiometric &  
Conductometric measurements**

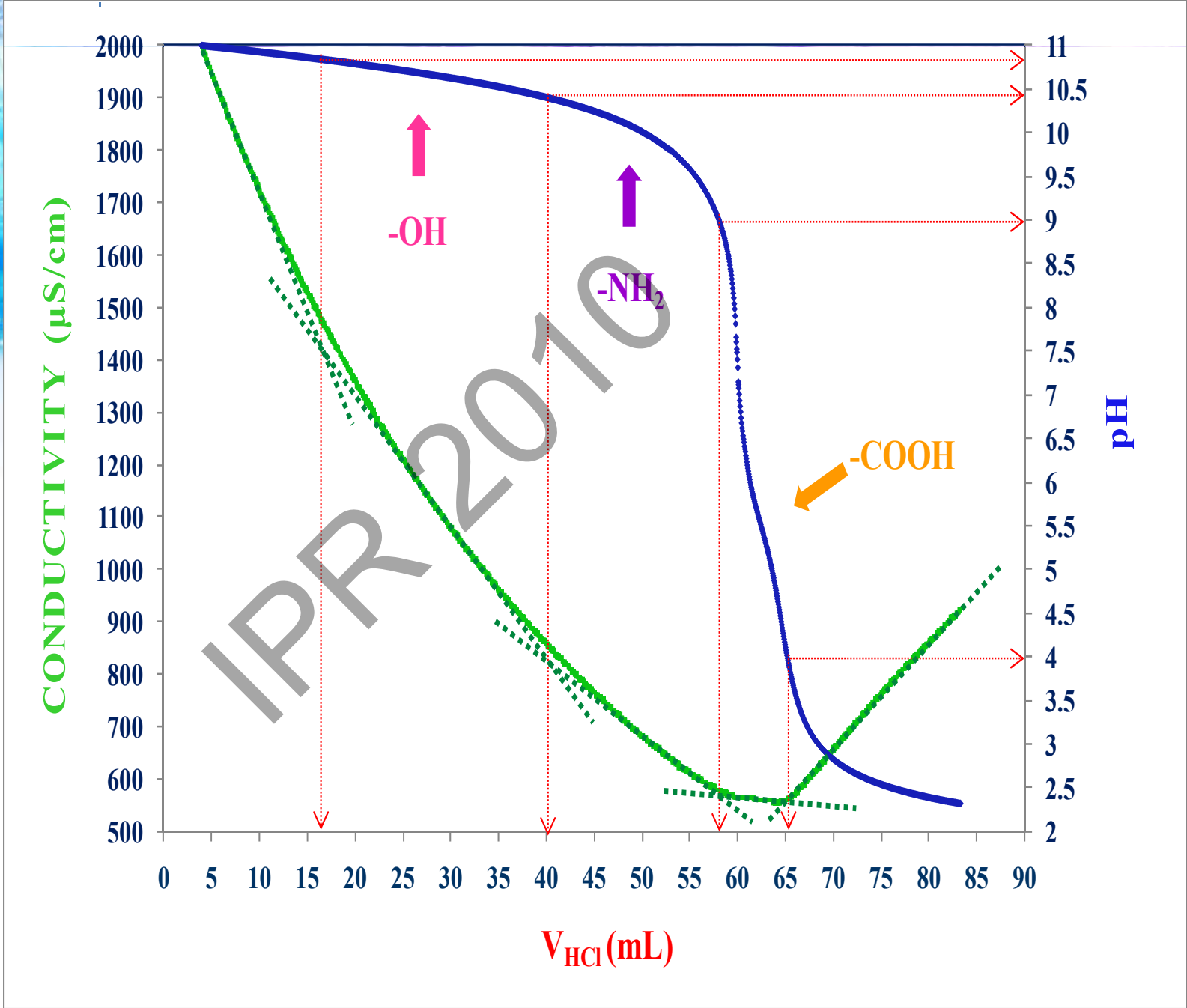
**Dynamic Light Scattering**

**Zeta Potential measurements**

# POTENTIOMETRIC & CONDUCTOMETRIC TITRATION

- Acid/Base Titrations
- Simultaneous measurement of pH & Conductivity





# DYNAMIC LIGHT SCATTERING

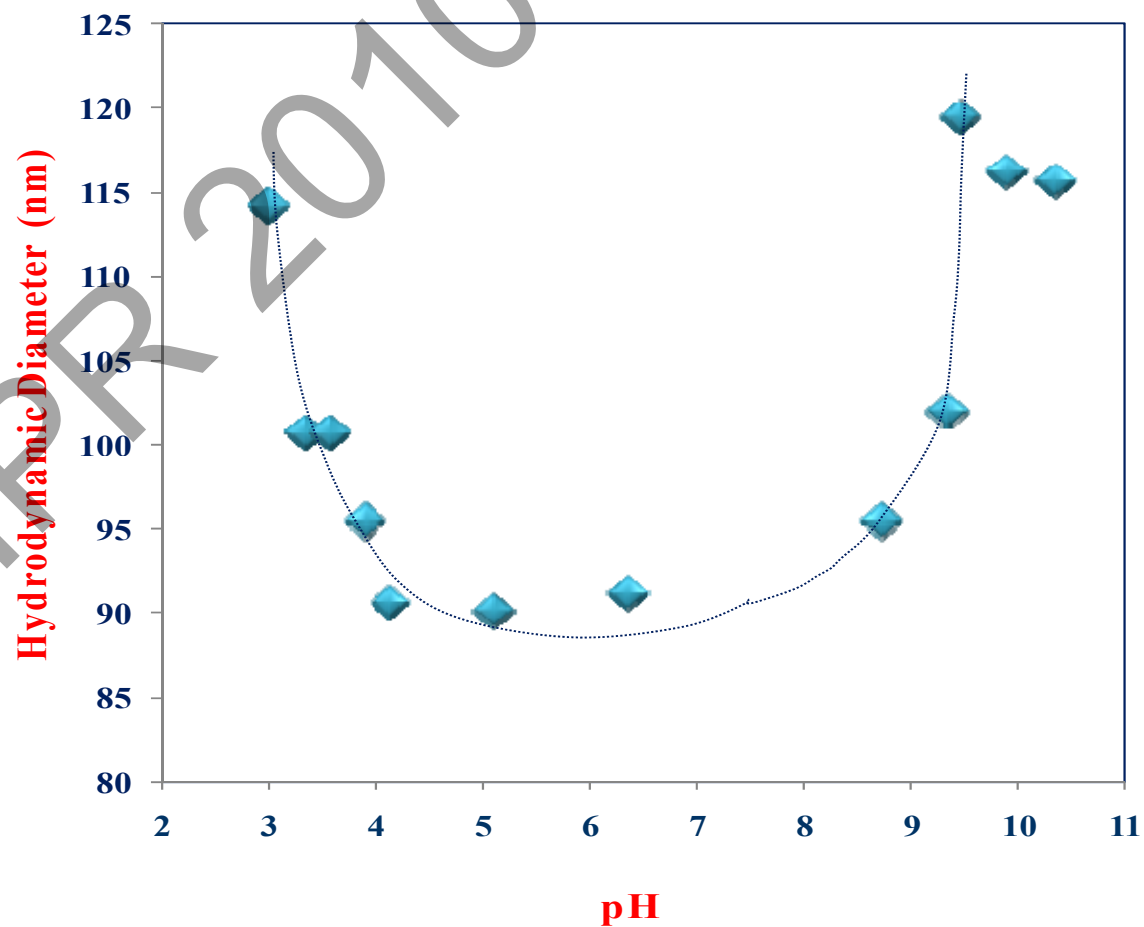
- Principle of Light Scattering
- Hydrodynamic Radius



Iso - electric point  
pH range 4 ~ 9

Maximum swelling  
~ 25 nm

Swelling in a very  
narrow range of pH  
3 ~ 4 & 9 ~ 10



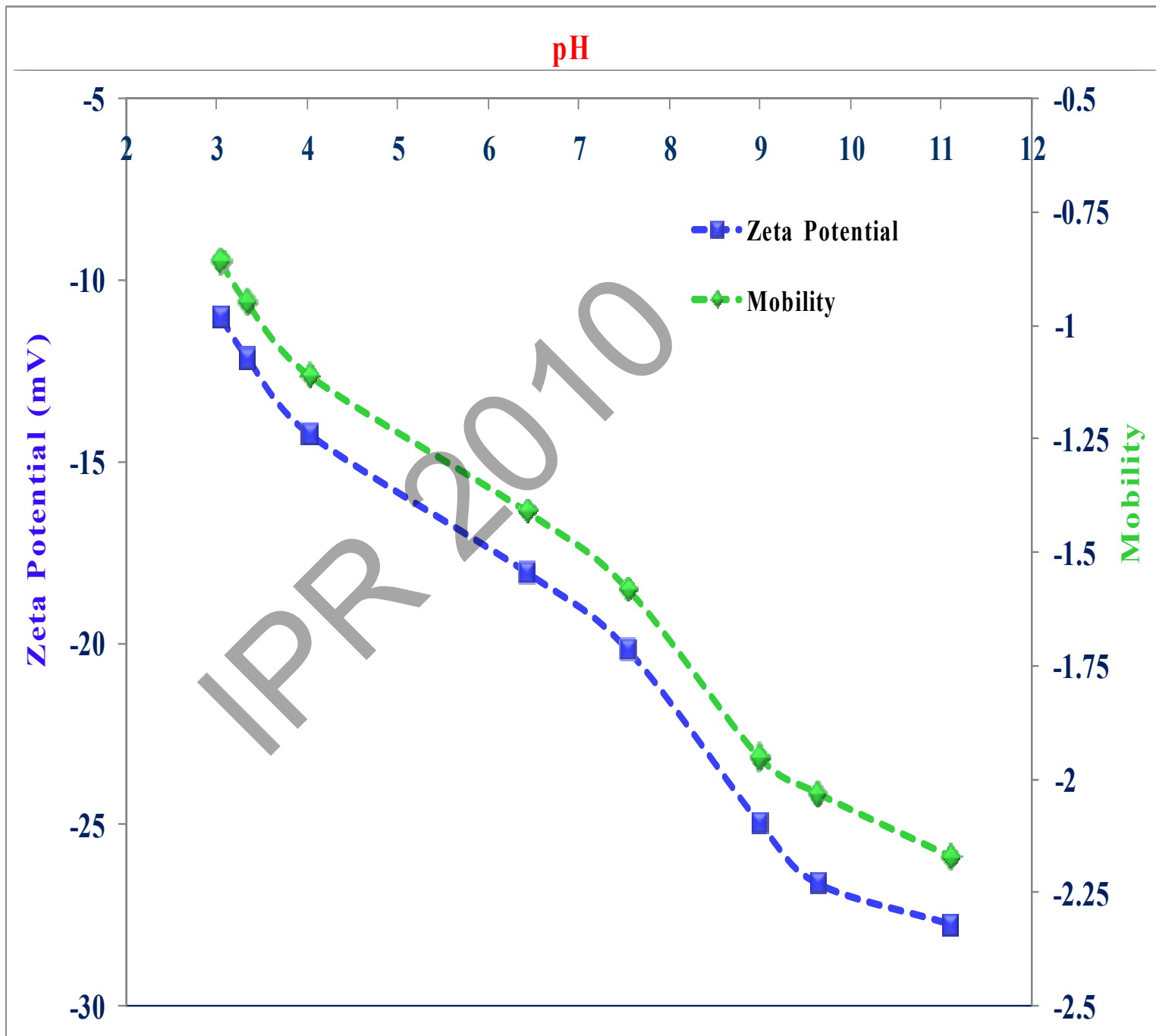


## ZETA POTENTIAL MEASUREMENTS

- Potential difference between the medium and the stationary fluid layer around the particle
- Electrophoresis



Image from <http://www.nbtc.cornell.edu/facilities/tools/Nano-ZS%20image.bmp>





## **NANO CRYSTALLINE CELLULOSE**

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# INTRODUCTION

Obtained from wood or cotton

Acid hydrolysis with sulphuric acid

Rod-like crystals

$L = 200 \text{ nm}$ ,  $D = 10 \text{ nm}$

Biodegradable & Sustainable

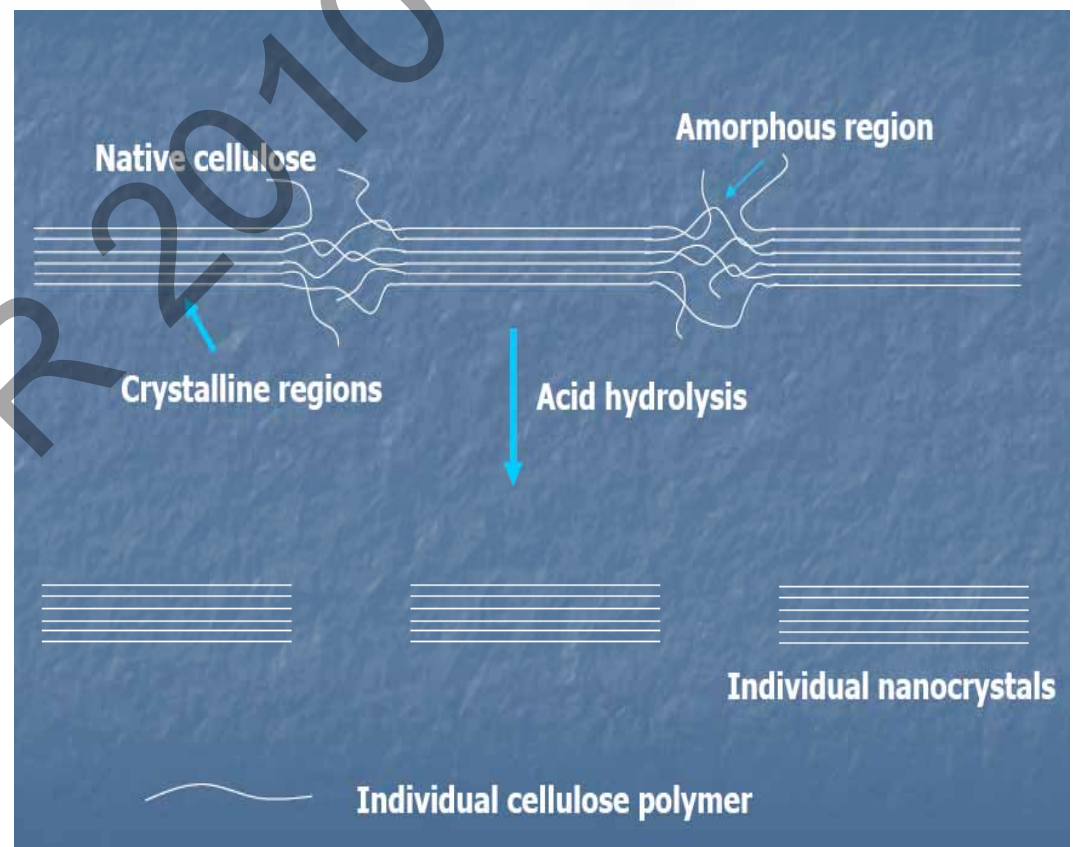
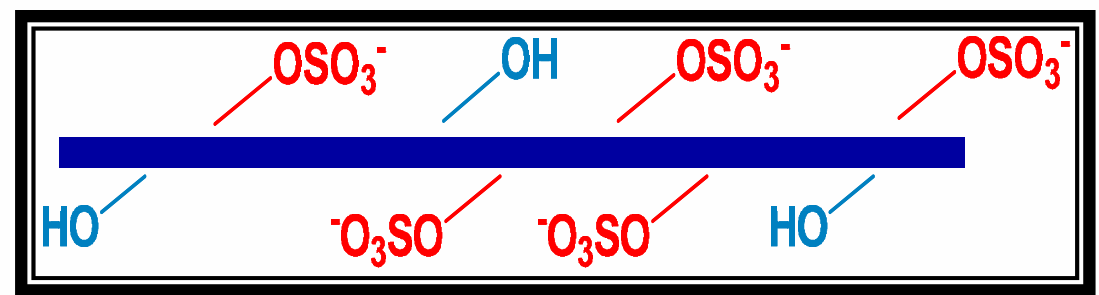
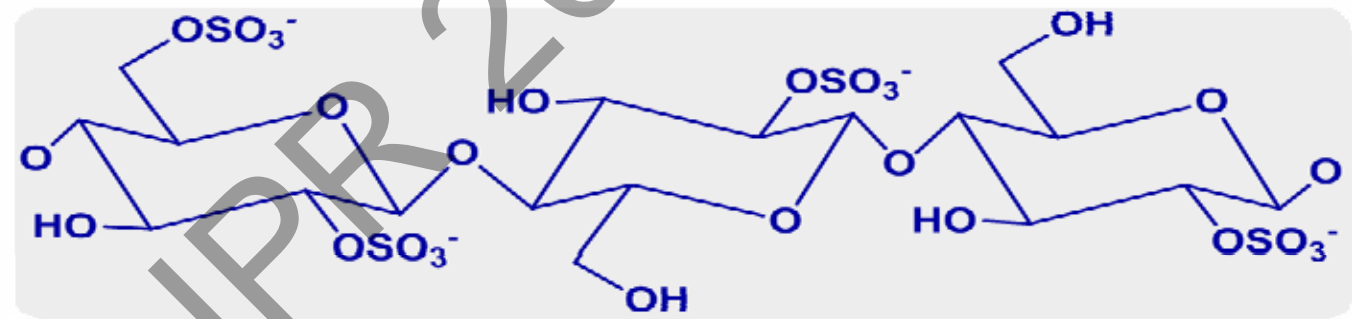
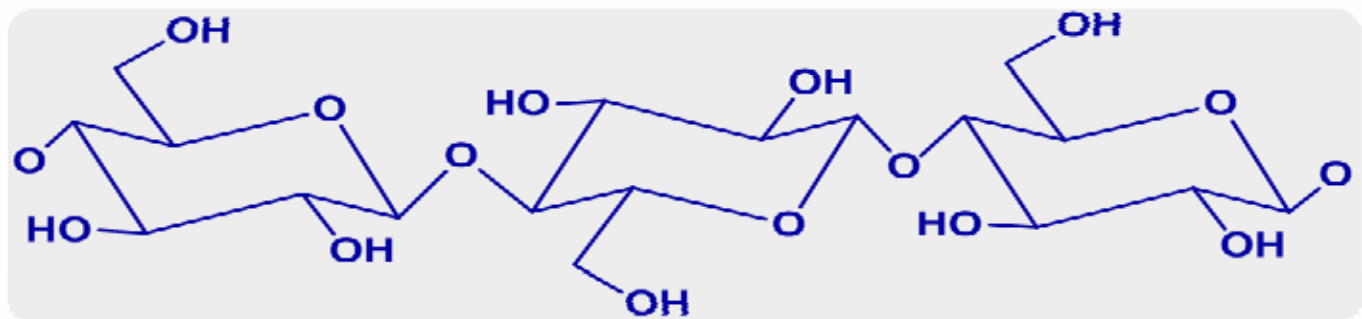


Image from <http://forestproducts.orst.edu/faculty/simonsen/Nanocomposites.pdf>



# BINDING INTERACTION WITH SURFACTANT

FOR POTENTIAL PERSONAL HOME CARE APPLICATIONS

Isothermal  
Titration  
Calorimetry

• Binding  
interaction in  
bulk

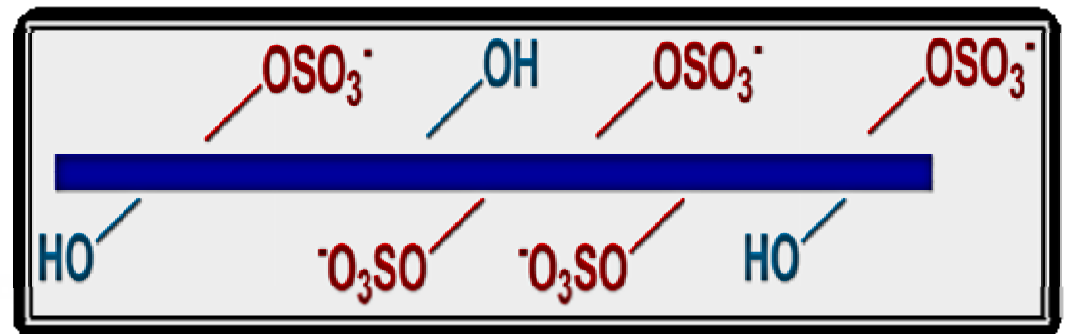
Tetradecyl Trimethyl Ammonium Bromide



Surface  
Tensiometry

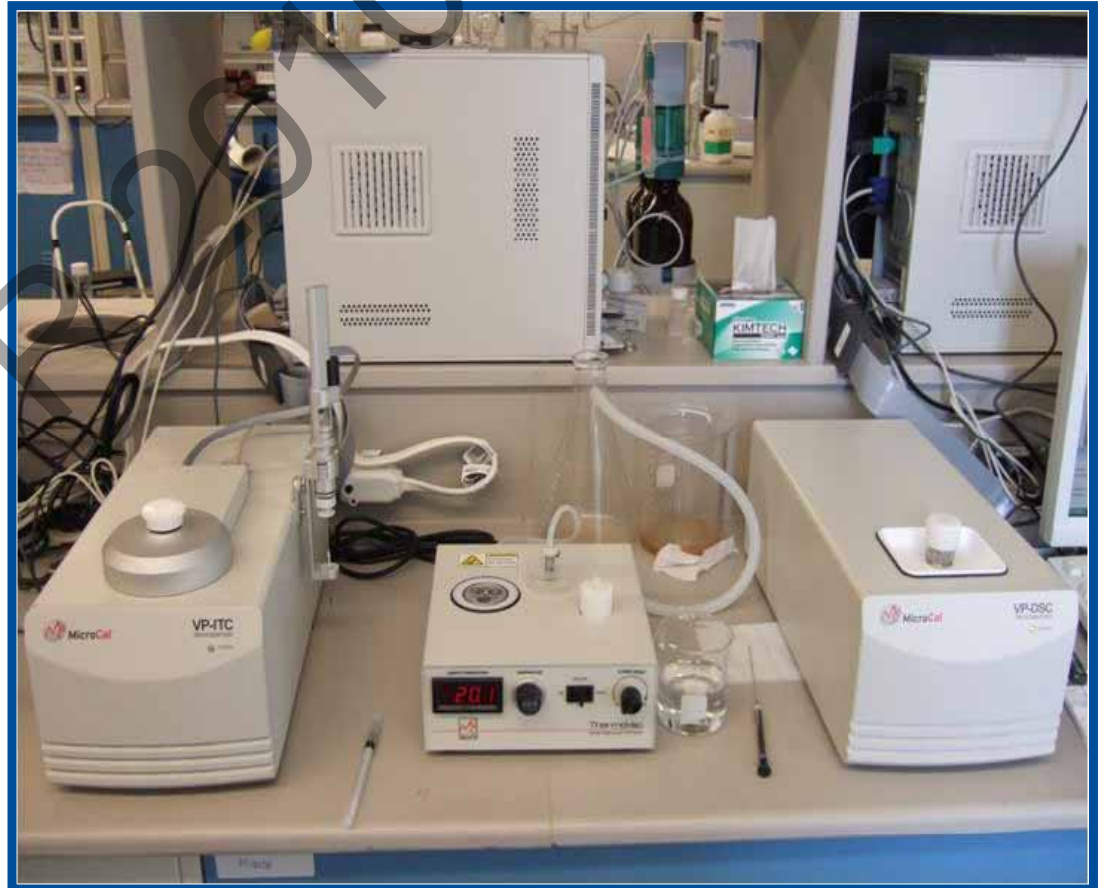
• Interaction at  
liquid-air  
interface

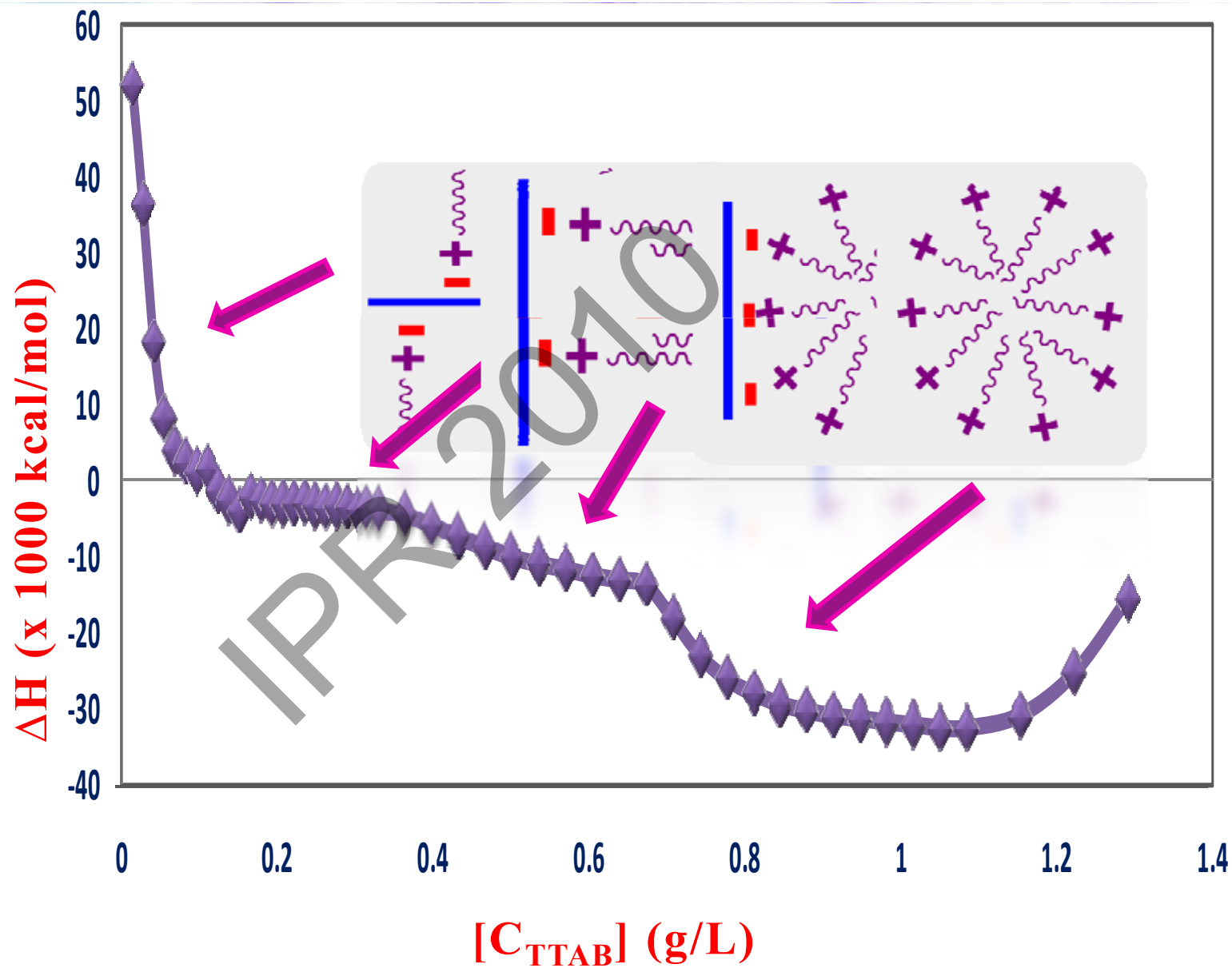
Nano Crystalline Cellulose



# ISOTHERMAL TITRATION CALORIMETRY

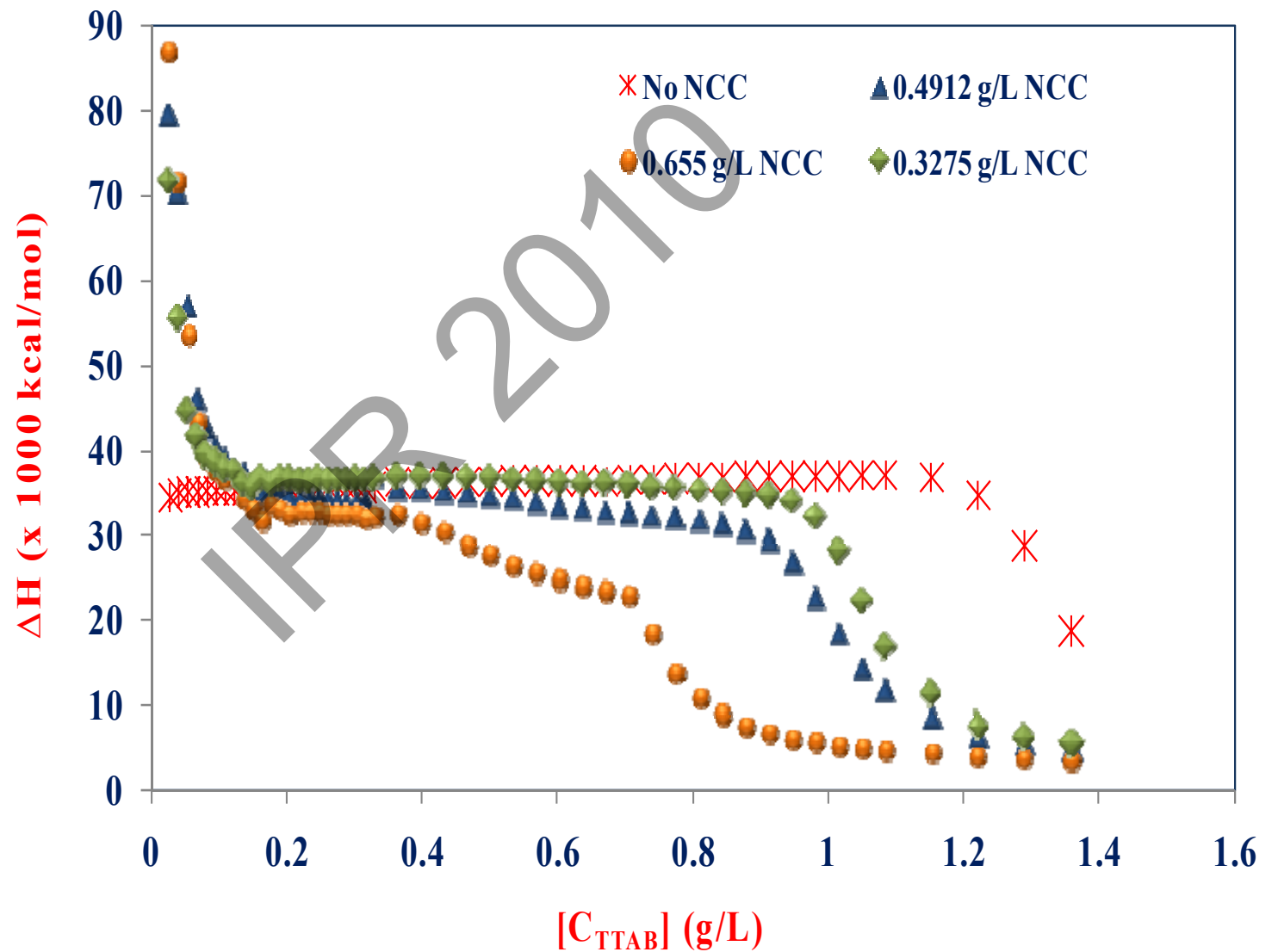
- Thermodynamic heat changes during chemical interactions
- Binding of polymer and surfactant







## EFFECT OF NCC CONCENTRATION ON ITC CURVES

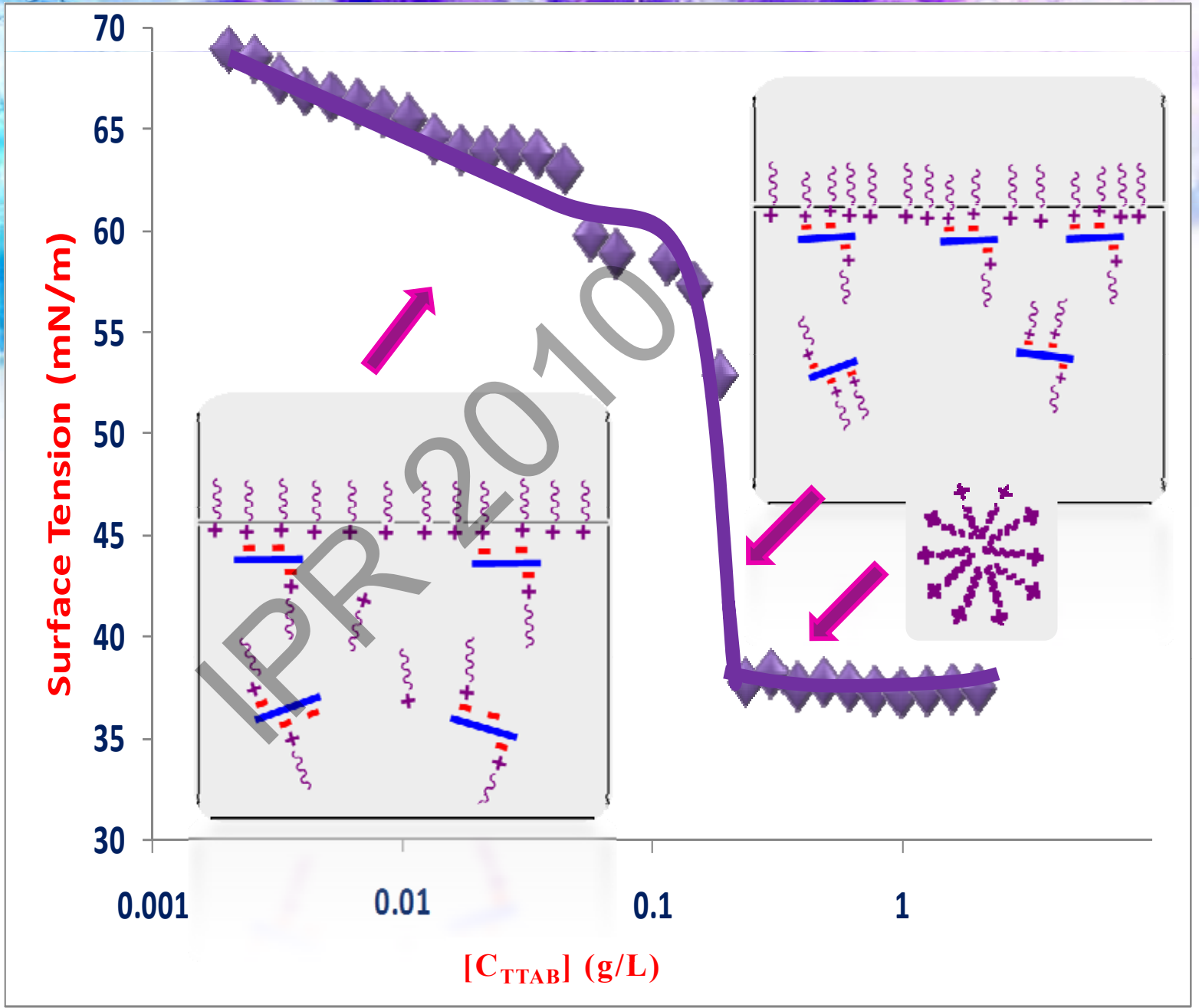


# SURFACE TENSIONOMETRY



➤ **Wilhelmy plate method**

➤ **Computer controlled dosing devices for precise titration of surfactants**

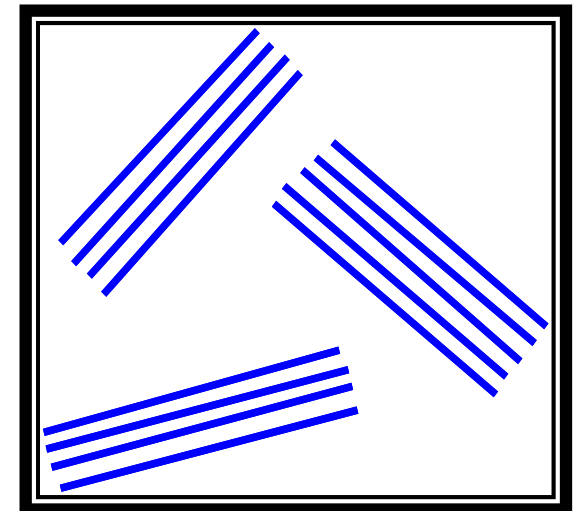
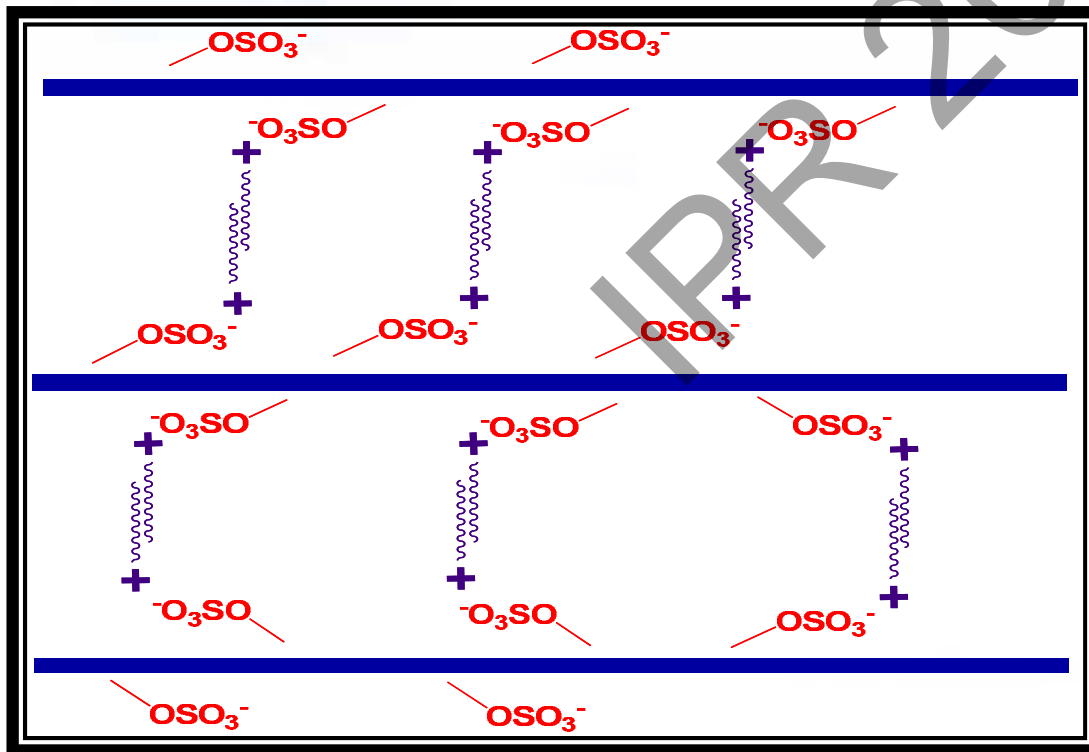


## PHASE SEPARATION IN PRESENCE OF SURFACTANT

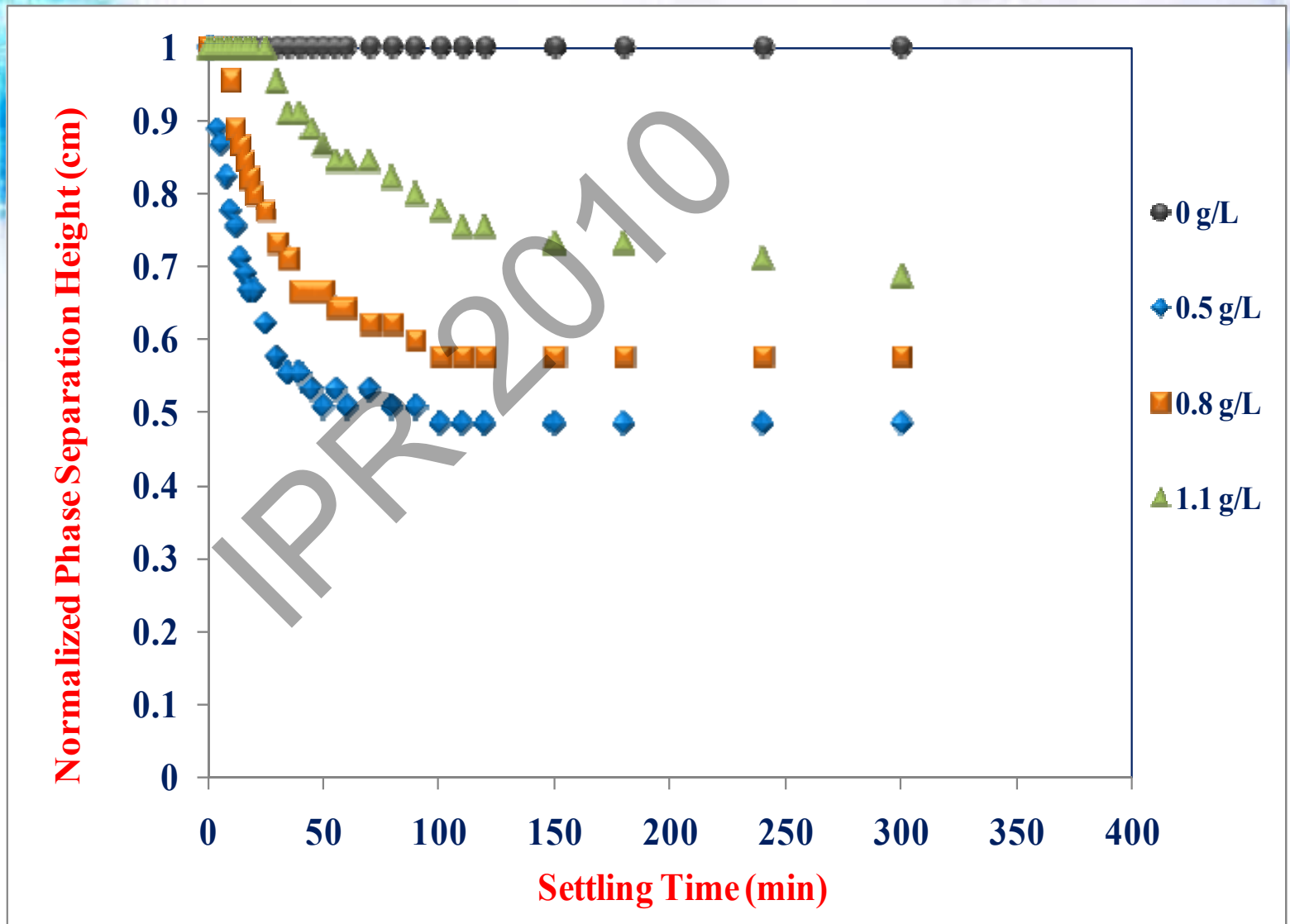


## STACKING UP OF NANOCRYSTALS

## FORMATION OF HYDROPHOBIC FLOCKS

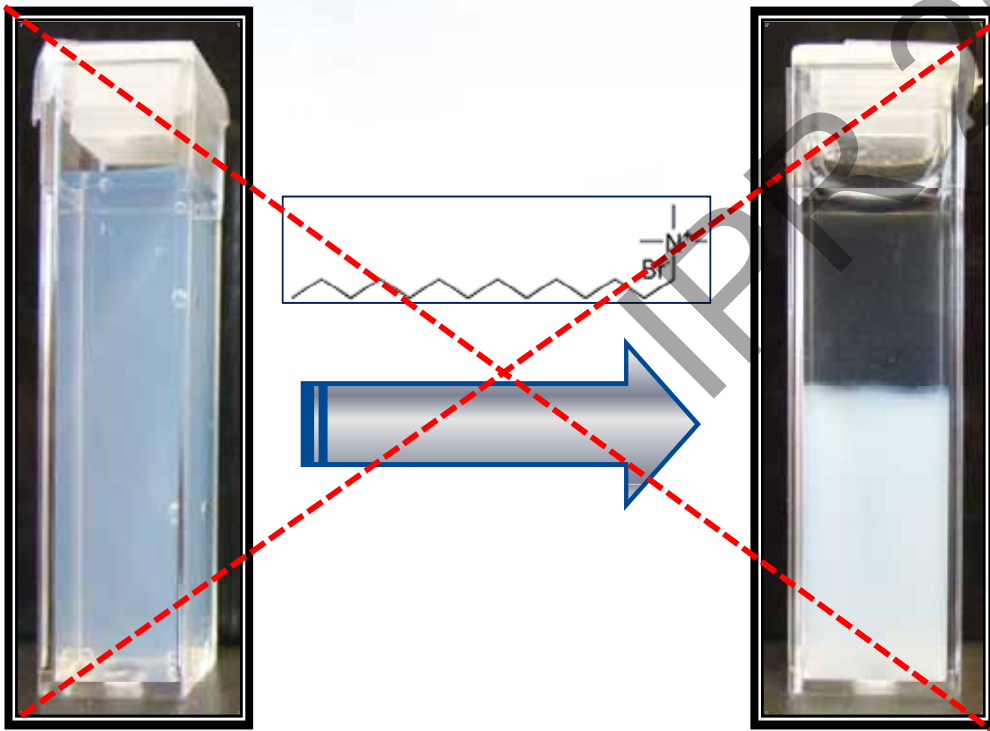


## MEASUREMENT OF PHASE SEPARATION HEIGHT

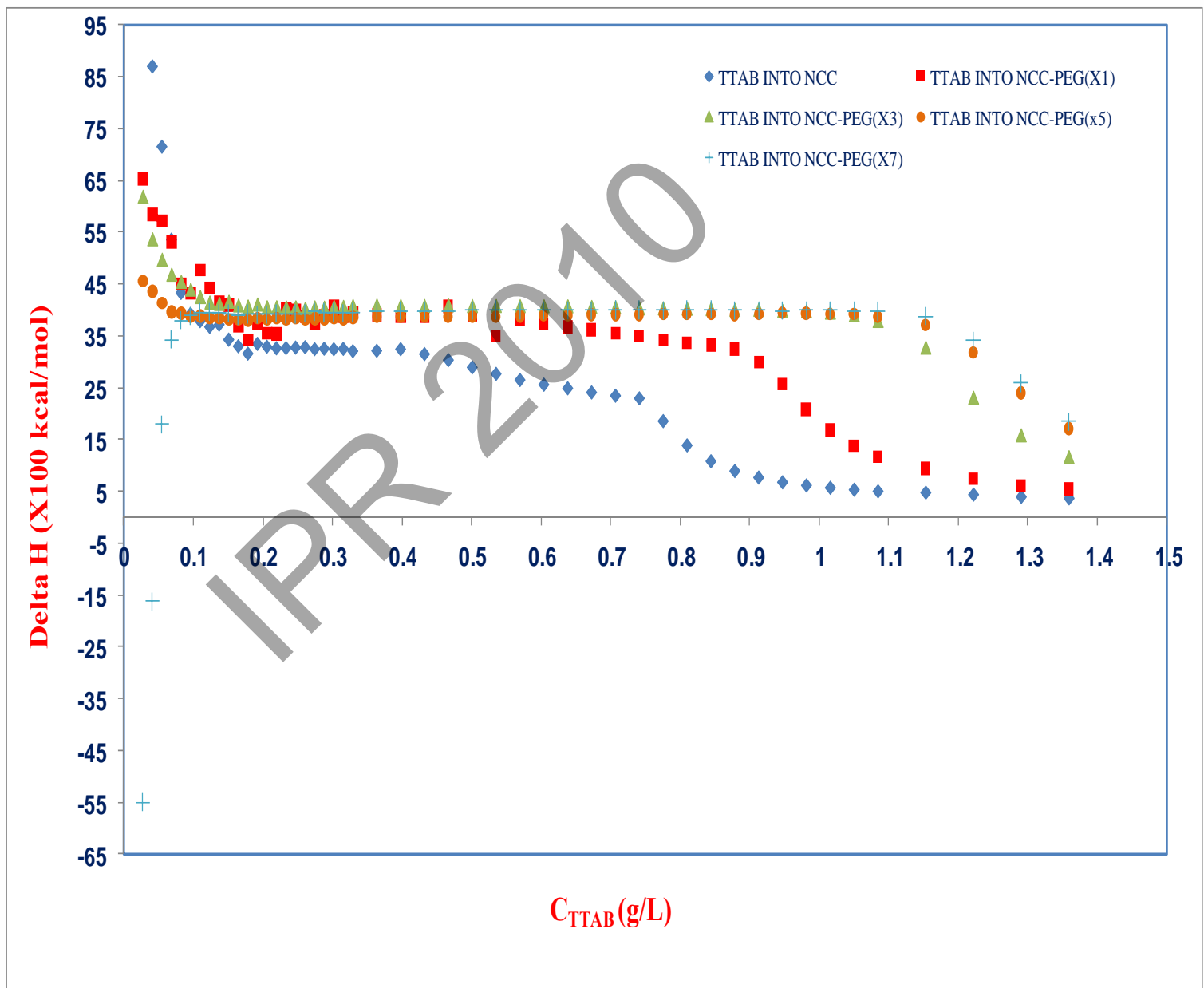


## GRAFTING PEG ON NCC SURFACE

- Steric stabilization of nanocrystalline cellulose by grafting PEG-2000, producing stable aqueous suspensions in presence of an oppositely charged surfactant
- Prevent formation of hydrophobic flocks & consequent phase separation



## ITC RESULTS FOR PEG GRAFTED NCC







## FUTURE WORK

- ❑ **Synthesis of robust polyampholyte microgels using modified PVA as crosslinker**
- ❑ **Incorporation of methylcellulose in the synthesized microgel particles**
  - **pH & temperature responsive properties**
- ❑ **Steric stabilization of nanocrystalline cellulose by grafting PEG-300**
  - **Prevent formation of hydrophobic flocks & consequent phase separation**



## SUMMARY

- ❑ **Cellulose based Polyampholyte Microgels have been synthesized using inverse microemulsion polymerization**
  - **Microgels show swelling at high and low pH & deswelling at IEP**
- ❑ **Binding interactions of nanocrystalline cellulose with an oppositely charged surfactant has been studied using various techniques**
  - **Three kinds of polymer-surfactant interactions are observed:  
Electrostatic binding, polymer induced micellization and hydrophobic interactions**

**THANK YOU**

