Rheological behaviour of starch (maize) in presence of surfactant medium

Thesis submitted

by

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National Institute of Technology

Rourkela

CERTIFICATE

This is to certify that the thesis entitled, **“Rheological behaviour of starch(maize) in presence of surfactant medium”** submitted by Sai Sankar chokkapu in partial fulfillments for the requirements for the award of Bachelor of Technology Degree in Chemical Engineering at National Institute of Technology, Rourkela (Deemed University)
Is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/ Institute for the award of any Degree or Diploma.

Date : 09/05/2014

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Department, NIT Rourkela.

Date: 09/05/2014 Sai Sankar Chokkapu

Contents

ABSTRACT

This work studies rheological behaviour of maize starch. Starches are biodegradable and cheap in cost and have many applications like Paper making, Corrugated board adhesives, Clothing starch, Construction industry, Noodles, Sauces, Meat products etc. Effect of different parameters like starch concentration, surfactant concentration are considered for this study. The effect of cationic cetyltrimethyl ammonium bromide (CTAB) and anionic surfactant sodium dodecylbenzenesulfonate (SDBS) on the rheological behaviours of starch solution were investigated. It is observed that viscosity increases non linearly with increase in concentration. Initially it increases slowly and it reaches to maximum at 37 wt% of corn starch. For CTAB with the increasing concentration of the surfactant viscosity increases up to certain point and then starts decreasing sharply. For SDBS it is observed that, at very low surfactant concentrations, there is a small increase in viscosity and it reaches maximum further there is almost no change with increasing SDBS surfactant concentration. Zeta potential values of corn starch in the presence of different concentrations CTAB and SDBS were investigated.

Key words: Rheology, Starch, Surfactant, Viscosity.

List of figures

Chapter1:

Introduction:

1.1

Rheology is the study of the flow of matter, primarily in the liquid state, but also as 'soft solids' or solids under conditions in which they respond with plastic flow rather than deforming elastically in response to an applied force. It is applicable to substances which have a complex microstructure, such as muds, suspensions, polymers and other glass formers(*e.g.,* silicates), as well as many foods and additives, bodily fluids (*e.g.,* blood) and other biological materials or other materials which belong to the class of soft matter.

Newtonian fluids can be defined by a single coefficient of viscosity for a specific temperature. Although this viscosity will vary with temperature, it does not vary with the strain rate. Only a few group of fluids exhibit such constant viscosity, and these are called as Newtonian fluids. But for an enormous class of fluids, the viscosity varies with the strain rate are called non-Newtonian fluids.

Rheology generally chronicle for the behaviour of non-Newtonian fluids, with rate of change of strains or strain rates one can characterize the minimum number of functions that are needed to relate stresses. For example, one cannot reduce viscosity of water by shaking but one can reduce the viscosity of ketchup by shaking (or other forms of mechanical agitation, where the relative movement of different layers in the material absolutely causes the reduction in viscosity). Ketchup is a shear thinning material, as aggrandize in relative velocity caused a reduction in viscosity, but some of the other non-Newtonian materials show the opposite behaviour. For shear thickening or dilatant materials viscosity increases with relative deformation.

The rheological behavior of starches(ex: maize,potato) and characterization of starches has received widespread attention in present years because of their broad range of industrial applications and academic interest. Surfactants are one of the indispensable components in cosmetics, gels, foods, etc. The presence of even small amounts of surfactants can differ the viscosity or rheological properties of the fluids.

Non-Newtonian Fluids:

If the relationship S/R is not constant then the fluid is said to be non-Newtonian fluid. For non-Newtonian fluids viscosity changes as the shear rate is varied. Thus, the parameters of spindle, viscometer model and rotational speed have a consequence on the measured viscosity. This measured viscosity is called apparent viscosity and is accurate when unambiguous experimental parameters are attached to. There are different types of non-Newtonian flow behaviour, characterized according to variations in shear rate by the way a fluid's viscosity changes in response.

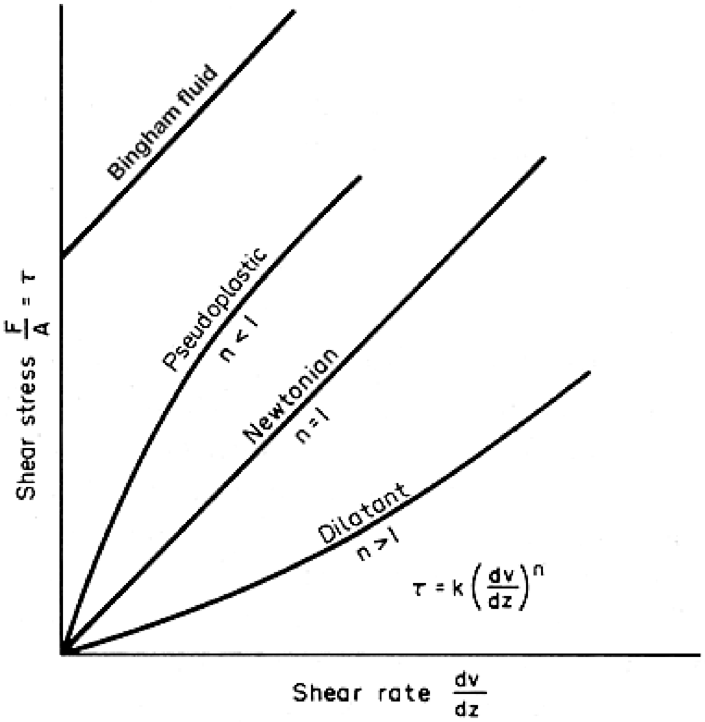
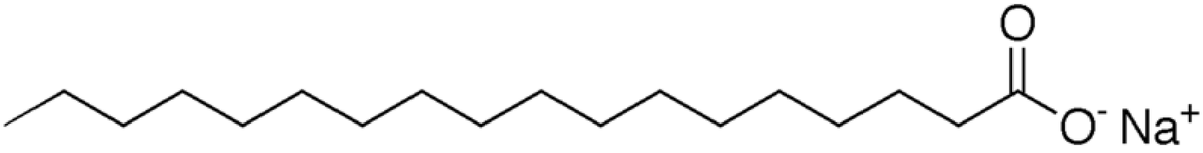


Figure.1 Shear Stress vs. Shear Rate for different fluids

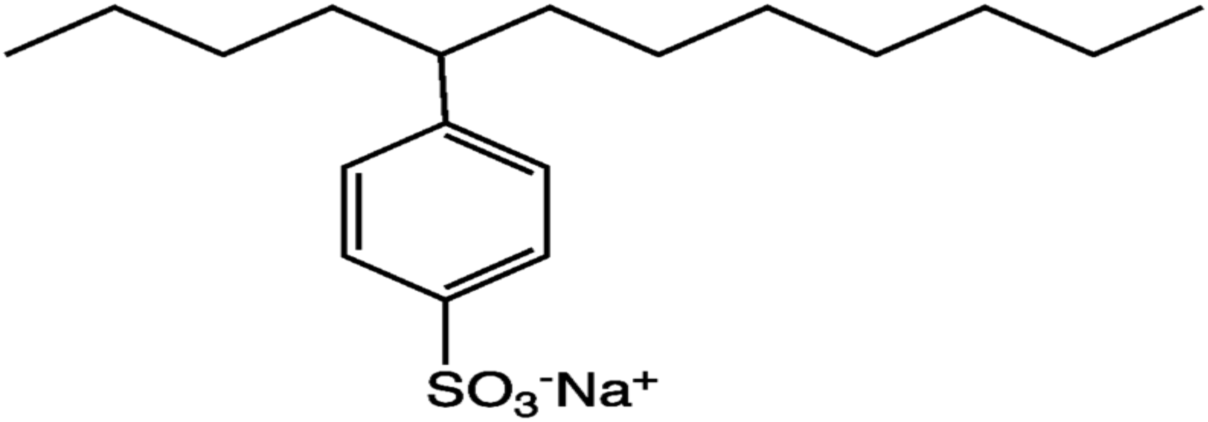
The knowledge of rheological properties for different materials for slurry preparation is of great importance. These topics are studied and severely important for several technical applications e.g. process control in chemical engineering, casting of ceramics, storage, transport of solids in pipelines and atomization.

1.2 Surfactants

Surfactants are compounds that lower the surface tension (or interfacial tension) between two liquids or between a liquid and a solid. Surfactants may perform as detergents, wetting agents, foaming agents, emulsifiers and dispersants.ex: sodium stearate used in soap



Sodium stearate



sodium dodecylbenzenesulfonate

1.2.1Types of Surfactants

There is a wide range of different surfactant types, each having solitary properties and characteristics. Depending on the category of the charge of the head, a surfactant belongs to the cationic, anionic, non-ionic or amphoteric/zwitterionic family.

A) Anionic surfactants

The head is negatively charged in the solution. For dishwashing liquids, laundering, and shampoos this is the most widely used type of surfactant because of its incompetent cleaning properties. The most commonly used anionic surfactants are alkyl ethoxylate sulphates, alkyl sulphates, and soaps.

B) Cationic surfactants

The head is positively charged in the solution. There are three different types of cationics each with their specific application:

Cationic surfactants provide softness in fabric softeners and in detergents with built-in fabric softener. Their main use in laundry products is in rinse added fabric softeners, example esterquats, which is one of the most widely used cationic surfactants in rinse added fabric softeners. In laundry detergents, at the stain/water interface cationic surfactants (positive charge) improve the packing of anionic surfactant molecules (negative charge).

This helps to reduce the dirt/water interfacial tension in a very effective way, leading to a more vigorous dirt removal system. They are especially effective at removing greasy stains. Mono alkyl quaternary system is an example of a cationic surfactant used in this category. In bathroom cleaners and household, cationic surfactants afford to the disinfecting/sanitizing properties.

C) Non-ionic surfactants

These surfactants do not have any electrical charge, which makes them resistant to water hardness deactivation. Non-ionic surfactants are excellent grease removers that are used in household cleaners, laundry products and hand dishwashing liquids.

Both non-ionic and anionic surfactants are mostly present in laundry detergents as they companion each other's cleaning action. Non-ionic surfactants afford to making the surfactant system less sensitive to hardness. Ethers of fatty alcohols are the most commonly used non-ionic surfactants.

1.3 Applications:

Starch has many industrial application:

1. Paper making.
2. Corrugated board adhesives.
3. Clothing starch [it is a liquid that is prepared by mixing a vegetable starch in water and is used in laundering of clothes].
4. Construction industry, where starch is used in the gypsum wall board manufacturing process.

Food applications:

1) Snacks
2) Baby food
3) Noodles
4) Sauces
5) Meat products.

Unlike other physical properties, such as thermal conductivity, heat capacity or density, which can be considered approximately constant during processing for a specific formulation, the apparent viscosity can greatly vary due to the effects of processing variables and changes occurring in the sample.

Dormant replacements for synthetic films in food packaging applications can be done using biopolymer films in acknowledgement to a strong marketing inclination towards more environmentally friendly materials. Starch has acquired considerable attention because of its totally biodegradable attribute and low cost. It is an appurtenant matrix-forming material and is also the most regularly used agricultural commodity, relative inexpensive with behold to other biopolymers. The dormant functional and nutritional properties of such edible films, in conjunction with their improved biodegradability contrast to other polymer systems, could certainly guarantee increased contemplation in the immediate future.

The starch films overall performance and coatings is highly inclined to be customizable, because of the attainability of a wide types of starches and their capacity for physical and chemical modifications. The hydrophilic nature of starch is a major constraint that seriously limits the development of starch-based materials; in fact, their properties depend on the ambient humidity .An alternative to reduce these drawbacks is the use of modified starches.

To produce low water sensitive materials (Fringant, Rinaudo, Foray, & Bardet, 1998) Chemical derivatization has long been studied as a way to solve this problem. Chemical changes includes the introduction of functional groups into the starch molecule using reactions of derivatization (etherification, cross-linking and grafting) or reactions of decomposition (acid or enzymatic hydrolysis and oxidation). This is a useful preference that allows to change the structure and accordingly the properties of native starches. The use of chemically changed starches to obtain films; predominantly substituted as well as pregelatinized starches were reported by assorted authors.

In many instances, the root and tuber starches contain significant amounts of mono phosphate esters covalently bound to starch. Potato starch is unique in comparison to other cereal starches (corn, wheat, rice, etc) because of its larger granule size, longer amylose and amylopectin chain length, presence of phosphate ester groups on amylopectin, ability to exchange certain cations with corresponding effects on viscosity behaviour, ability to forma thick visco-elastic gel upon heating and subsequent cooling in water, and poor thermal and shear stability of this gel.

The use of starch in various products and manufacturing processes is determined by its functional properties such as gelatinization, pasting, retrogradation, viscosity, swelling and solubility, water absorption which vary considerably from crop to crop and with ecological and agronomic influences. The starch functional properties are dependent on composition and molecular structures of the starches which include amylose/amylopectin ratio, phosphorus content, granular size, and molecular weight of the starches and chain length distribution of amylopectin.

Chapter2:

Literature review:

The rheology of starch and their characterization has received broadspread attention in recent years because of its broad range of industrial applications and academic interest. Viscosity or rheological properties of emulsion, or paste is also important in changing the physical properties of cosmetics, creams, foods, bio fluids etc. These fluids can be subjected to surfactant treatment to change the viscosity or rheological properties or these systems are containing surfactants as one of the essential components that may cause the change in rheological properties. Interactions between the particles in the starch paste are very important in the rheological behaviour of the system. In general, the properties depend on different parameters like solid/water ratio, size, shape, etc.

The most common example of a shear thickening system is corn starch and water. Investigations into the non-Newtonian flow behaviour of corn starch slurries have studied from elementary school demonstrations to in-depth rheological examinations that use corn starch to further elucidate the mechanisms that drive shear thickening. Here, we determine how much corn starch is required for the average person to ‘‘walk on water’’. Steady shear rate rheological measurements were engaged to monitor the thickening of corn starch slurries at concentrations ranging from 0 to 55 wt% (0–44 vol%). A transition from continuous to discontinuous thickening behaviour that exists at 52.5 wt% revealed by the shear rate ramp steady state experiments. Thickening behaviour in which was tested by dropping a 2.1 kg rock onto the suspension surface is done after comparing rheological data to macro-scopic pool experiments. Impact-induced thickening in the ‘‘rock drop’’ study was not noticed until the corn starch concentration approched at least 50 wt%. At 52.5 wt%, the corn starch slurry displayed true solid-like behaviour and the falling rock ‘‘bounced’’ as it impacted the surface. The corn starch pool studies were fortified by steady state stress ramps which were extrapolated out to a critical stress value of 67,000 Pa (that is the force generated by an 80 kg adult while running). Only the suspensions containing at least 52.5 wt% (42 vol.%) thickened to high enough viscosities that could reasonably be believed to support the impact of a man’s foot while running. Therefore, we conclude that at least 52.5 wt% cornstarch is required to induce strong enough thickening behaviour to safely allow the average person to ‘‘walk on water’’.

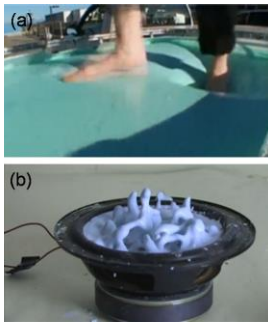


Figure.2 (a) Myth Busters walking on ‘‘water’’ YouTube video: Myth Busters – Walking on water – Ninja part 5/5) and (b) ‘‘Corn starch monsters’’ in YouTube video: Non-Newtonian fluid on a speaker cone.[1]

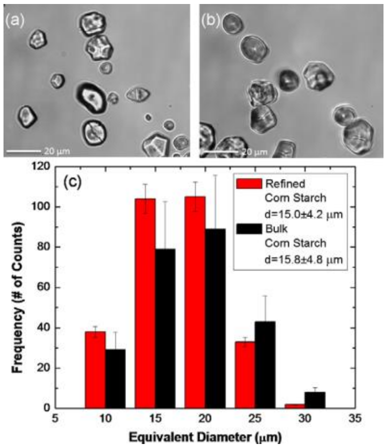


Figure.3 (a) Image of microscope refined and (b) bulk corn starch particles suspended in water.(c) Refined particle size distribution (red) and bulk (black) corn starches. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)[1]

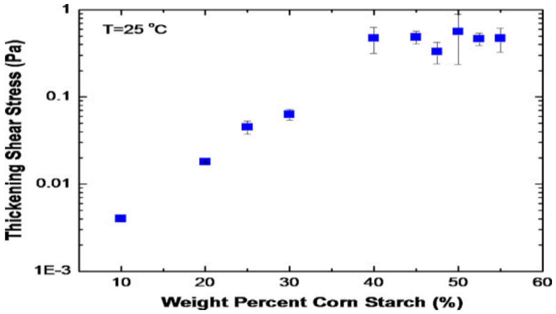


Figure 4. Thickening shear stress as a function of corn starch concentration (wt.%).

2.1 Studies on effect of surfactant:

The effect of starch composition and concentration on the rheological properties of starch in a mixed solvent, water–DMSO, was investigated in dynamic shear and extensional mode studied by B. Kapoor, M. Bhattacharya (2000). 70% amylose and 30% amylopectin are constituents of High amylose corn starch, common corn starch containing 25% amylose and 75% amylopectin, and about 99% amylopectin in waxy corn starch were used in this study. Different concentrations of 2, 4, 6, and 8% (w/v) in 10% water-90% DMSO (v/v) were used for each starch type. A behaviour change from semi dilute solution to viscoelastic solid at a concentration of 8% (w/v) occurred when an increasing the amylopectin content of starch from 30 to 99%. At 2% of concentration, an increment in the amylopectin content of starch from 30 to 99% which shows a way to change from Newtonian to incipient gel-like behaviour. At intermediate concentrations with increasing amylopectin content, in between behaviour of 4 and 6% (w/v) varied from semi dilute to critical gel-like substance. We observe here a power-law relaxation for all concentrations of customary and waxy corn starches with the slope decreasing with increase in concentrations. A 2% high amylose corn starch solution displayed Newtonian behaviour, while a 2% waxy corn starch solution displayed extension thinning behaviour.

T.D. Karapantsios studied the rheological and physical characterization of pregelatinized maize starches. This work shows maize starches modified by a small-scale industrial double drum dryer. With respect to the morphology the impact of the processing thermal treatment is examined, colour and texture of the dry product sheets. To appraise the moisture exchange capability of the product sheets Moisture absorption/desorption tests are employed and also construe information about its bulk porosity. The variation of the intrinsic and apparent viscosity of the pre gelatinized starches reconstituted with water to pastes were scrutinized when the drum speed, steam pressure and level of the gelatinization pool between the drums are varied. It is inspected that the dispersed swollen starch granules jurisdiction the rheological behaviour of the starch pastes.

That native starch can be chemically modified to improve its functionality and to expand its uses studied by E. Zaritzky (2008). Modified starches were characterized and the rheological behaviour of filmogenic suspensions was analyzed. The film forming capacity of different chemical modified corn starches were investigated. Since their films exhibited the lowest water vapour permeability acetylated starch was selected by the characteristics of the resulted films; its choicest concentration was 5% w/w. On film properties the repercussion of glycerol as plasticizer depend upon on its concentration, being 1.5% w/w those that allows to obtain the lowest WVP value, low film solubility in water and a more crowded structure than those of un plasticized films. Plasticized acetylated starch films mechanical behaviour achieve on glycerol concentration, being ascetic and brittle the un-plasticized ones, ductile those containing 1.5% w/w of glycerol and very conformable those with a higher plasticizer content.

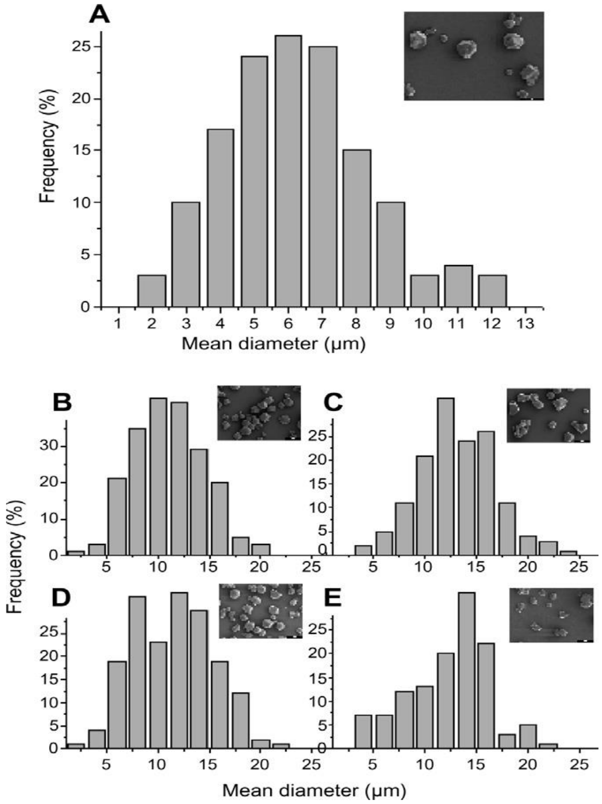


Figure 5. Histograms of granular diameters and micrographs of the different starches: (A) native starch; (B) acetylated starch; (C) acetylated cross linked starch; (D) hydroxypropylated cross-linked starch; (E) acid modified starch [5].

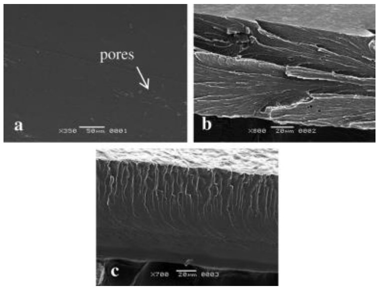


Figure 6. SEM micrographs of (a) surface and (b) cross-section of acetylated corn starch films. (c) Cross-section of acetylated corn starch plasticized with 1.5% w/w of glycerol. Films were formulated with acetylated corn starch at 5% w/w. Magnification is indicated in the micro graphs [5]

The effect of surfactants on the physical properties of corn starch films studied by Amparo chiralt(2013). He studied about rheological behaviour, particle size, zeta potential, surface tension and contact angle. The droplet size distribution, volume-length mean diameter and volume-surface mean diameter were determined in film forming dispersion, in triplicate, with a laser light scattering instrument. All film forming dispersion were diluted to a droplet concentration of 0.02% using deionized water to obtain z-potential values. Zeta Potential was determined at 25 degrees centigrade with Zetasizer nano-Z. To convert the electrophoretic mobility into z-potential values the Smoluchowsky mathematical model was used. The surface tension was determined at 25 degrees centigrade by means of the ring method using a ring tensiometer and a platinum-iridium ring of 19.09 mm in diameter. Measurements were performed in triplicate in each film forming dispersion.

2.2 Concluding remark:

Much of the work has been done on study of rheology of starch. The effects of cationic and anionic surfactants as well as polymers on the rheology of starches were seen in the various studies. The viscosity and zeta potential data was obtained for the starches and analysis had been done on the variations of the concentrations of the starches.

In this project the effect of surfactants on the viscosity of corn starch was studied. Starch has also a wide range of applications in industry paper making, Corrugated board adhesives, clothing starch, construction industry. Starch is one of the very conventional low costs natural material.

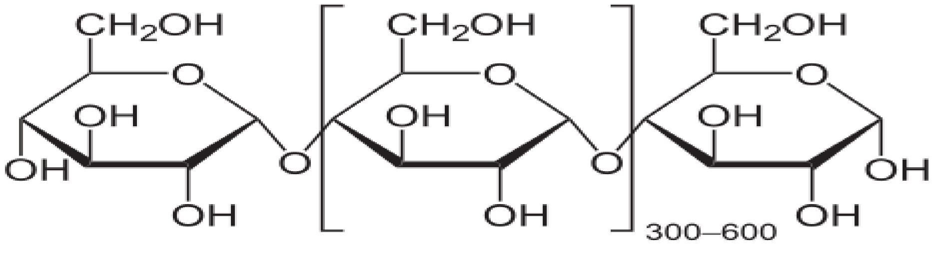


Figure 7. Structure of corn starch

2.3 Research objectives:

The objective of this project is to study the rheological behavior of starch. Effect of different parameters like starch concentration, pH, surfactant and electrolyte are considered for this study.

The specific objectives of this study are

- To study the rheological behaviour of starch (maize) for different starch concentration.
- To study the effect of shear rate on the viscosity of the starch solution.
- To study the effect of cationic surfactant (CTAB) on the viscosity of the starch.
- To study the effect of anionic surfactant (SDBS) on the viscosity of the starch
- To study the effect of surfactant concentration on the ζ potential of the starch.

Chapter 3

Materials and methods

3.1 Materials

Starches (maize) was purchased from Loba Chemie Pvt. Ltd., India and cetyltrimethyl ammonium bromide (CTAB) was purchased from Loba Chemie Pvt. Ltd., India. For measurement Ultrapure water of 18.2 MΩ.cm resistivity and pH 6.5–7 was used at 30 degrees centigrade for all the experiments.

3.2 Methods

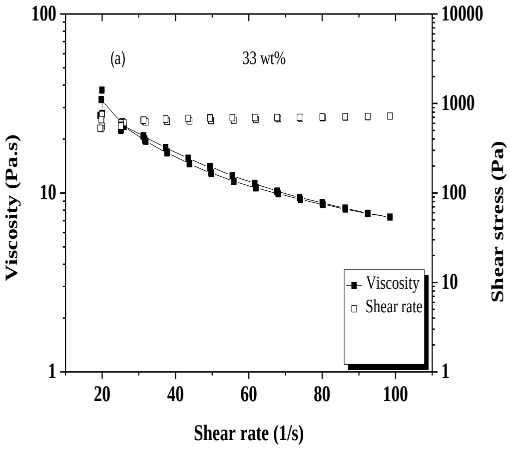
For viscosity measurement a rotational cone and plate BOHLIN VISCO-88 viscometer was used. The angle of the conical section was 5.4^0c and diameter 30 mm. A gap of 0.15mm was maintained between the cone and plate for every measurements .A cover was used with a wetted layer of sponge inside to decrease the moisture loss from the slurry during measurement. All the measurements were carried out at constant temp. 100C. The viscosity of the solution without additives containing 33%, 35%, 37%, 39%, 40% (% by wt.) were quantified at variable shear rates as well as at constant shear rates at 25 sec^{-1}, 31 sec^{-1}, 37 sec^{-1}, 43 sec^{-1}, 49 sec^{-1}. The slurry in presence of desired amount of CTAB and SDBS surfactant was prepared and viscosity of the samples were measured at constant shear rate37 $sec^{-1}$and at temperature of 100C.

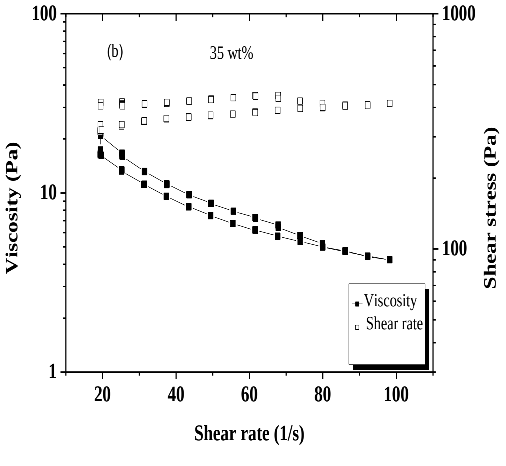
Chapter 4: Result and discussion:

Rheology is the study of the flow of matter, primarily in the liquid state, but also as 'soft solids' or solids under conditions in which they respond with plastic flow rather than deforming elastically in response to an applied force. Rheological behaviour of starches are important because they have vital applications.

4.1 Effect of starch concentration:

The rheological behaviour was studied for five dissimilar concentrations (33%, 35%, 37%, 39%, and 40% by wt) of cornstarch-water slurry, and the curves were obtained by changing the shear rate from 20 to 100 sec^{-1}. It is noticed that the shear stress increases with an increase in the shear rate and that the viscosity decreases with an increase in shear rate. Also, the upward curve cycle has almost equal viscosity when compared with the downward curve. The slurry shows non-Newtonian and dilatant (shear thickening) behaviour; that is viscosity is a function of the shear rate.





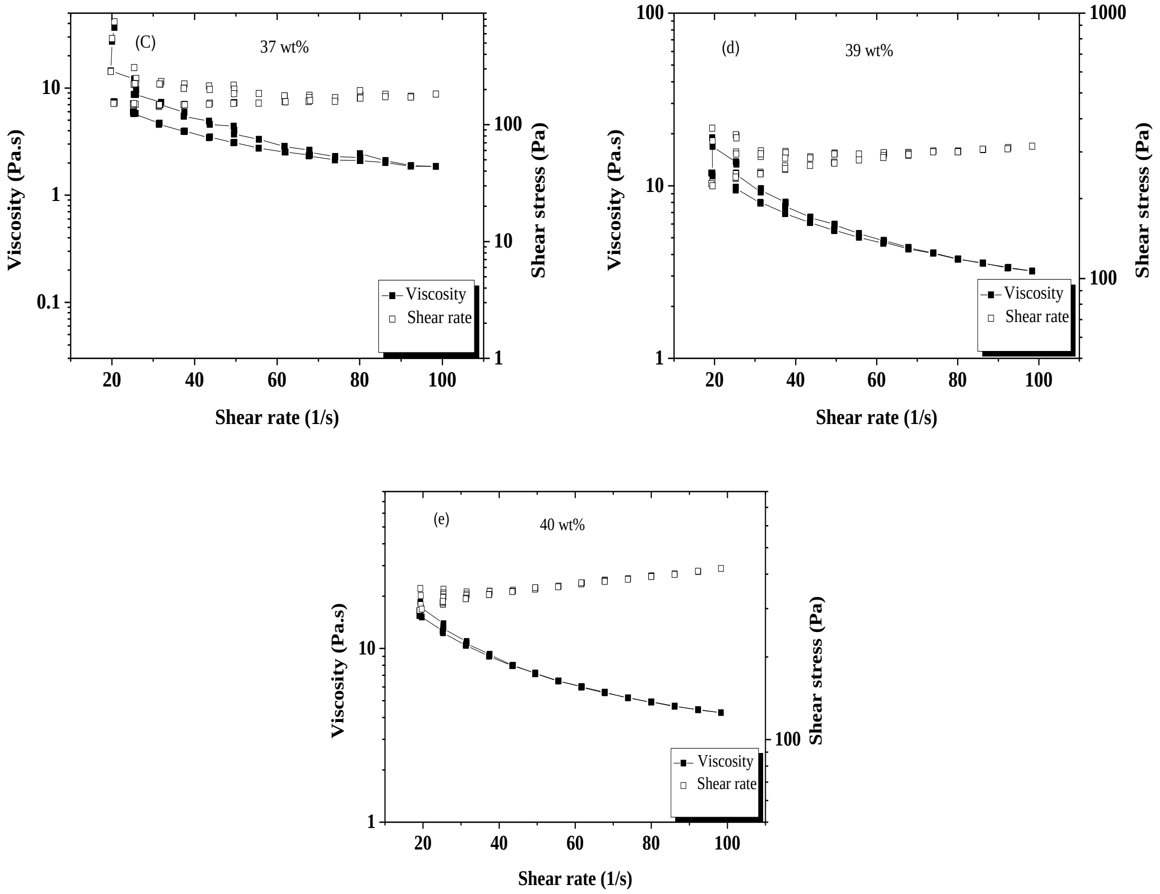


Figure 8. Shows variation of shear stress and viscosity with respect to shear rate.

Viscosities of different concentrations were plotted in figure 8 at 37 1/sec shear rate. It is observed that viscosity increases non linearly with increase in concentration. Initially it increases slowly and it reaches to maximum at 37 wt%.

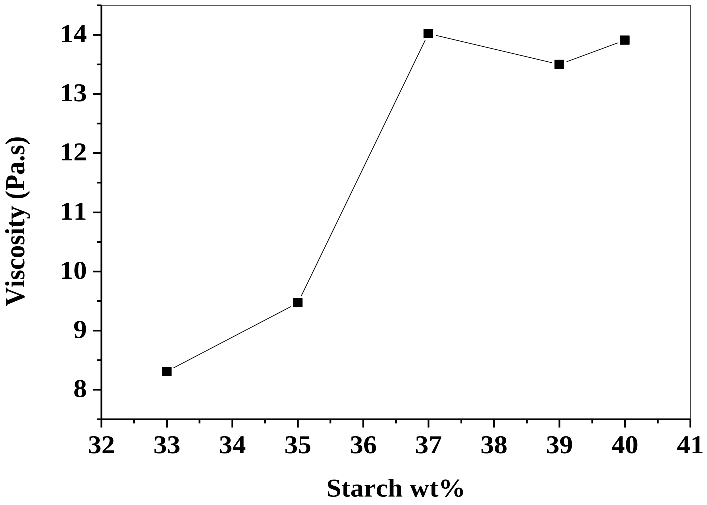


Figure 9. Variation of viscosity with respect to corn starch weight percentage

To know the behaviour of starch variation of viscosity with respect to shear rate is required. Fig 10 shows the viscosities obtained at constant shear of 25 1/s, 31 1/s, 37 1/s, 44 1/s, 49 1/s. For 37 starch wt% it is observed that as shear rate increases viscosity decreases.

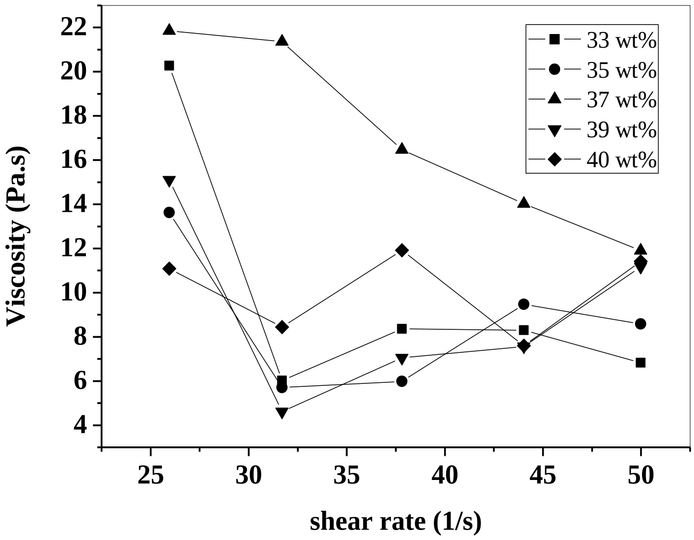


Figure 10. Variation viscosity with respect to shear rate.

4.2 Effect of Surfactants on Viscosity:

The viscosity of the slurry in the presence of all the surfactants with increasing concentration of the surfactants was observed at a constant shear rate of 37 s-1.

4.2.1 CTAB Surfactant:

It is noticed that, with the increasing concentration of the surfactant in the slurry, the viscosity increases up to 0.9 mM concentration (12.5 Pa.s) and then starts decreasing to a value of 10.3 Pa.s at 1 mM concentration. This behaviour may be elucidate in terms of the surfactant adsorption on oppositely charged surfaces. The zeta potential value of corn starch is negative so the starch is negatively charged, the cationic surfactant (CTAB) adsorbs in the begining on the surface using the head groups toward the solid surface, and the surface develop into hydrophobic. The hydrophobicity of the corn starch increases the coagulation tendency and finally shows higher viscosity. Further, with the increase in surfactant concentration, surfactants form a bilayer (hemimicelle) by attaching the tail groups toward the solid surface, and the surface becomes more hydrophilic.

SDBS Surfactant:

Various concentrations of SDBS were taken and the viscosity was measured at 37 1/s shear rate. Diversify in viscosity in the presence of SDBS is shown in the fig 11. It is observed that, at very low surfactant concentrations, there is an immature increase in viscosity slowly it reaches maximum further there is decrease in viscosity with increasing SDBS concentration. These values can be correlated with zeta potential results.

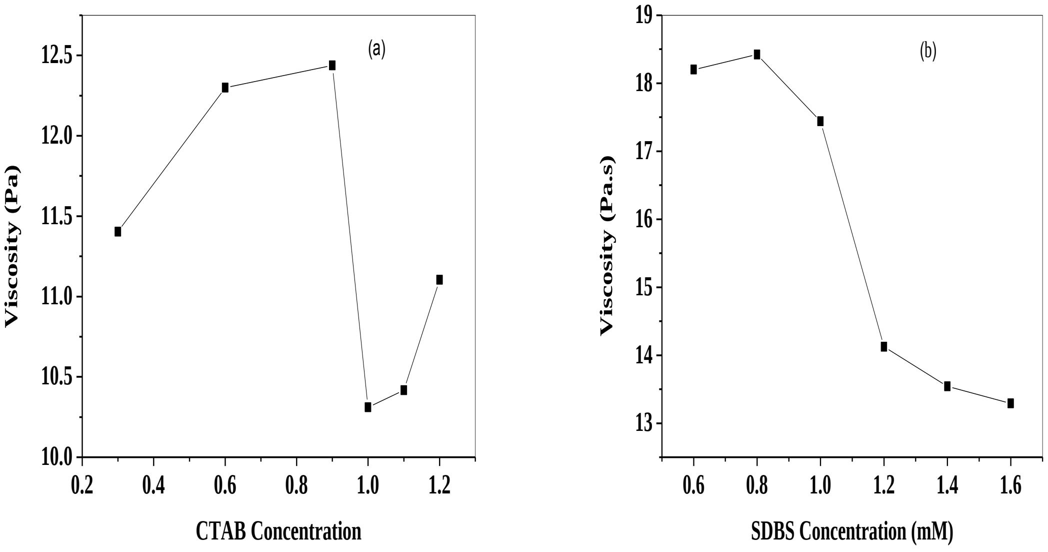


Figure 11. Variation viscosity with respect to surfactant concentration.

4.3 Zeta potential of starch particle

Zeta potential values of starch particle in aqueous medium and surfactant medium were investigated. In aqueous medium zeta potential value is -8.94 mV which shows that surface of starch particle in aqueous medium is negatively charged.

4.3.1 CTAB surfactant medium

Figure 12 Shows zeta potential variation in CTAB medium. Due to the presence of CTAB initially the cationic surfactant will be adsorbed through the head group and the surface become more hydrophobic due to the orientation of tail groups towards aqueous medium. With increase in CTAB concentration surface become more positive due to the development of bilayer of surfactant molecules.

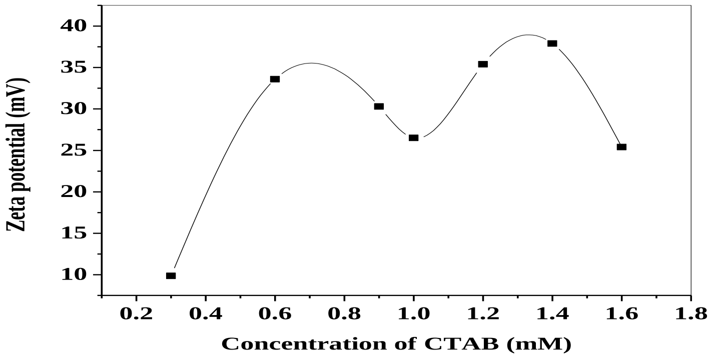


Figure 12. Variation of zeta potential with respect to CTAB concentration.

4.3.2 SDBS surfactant medium

Figure 13 Shows zeta potential variation in SDBS medium. In presence of SDBS the zeta potential is more negative than in presence of pure water due to adsorption of anionic surfactant molecules at the corn starch surface through tail groups.

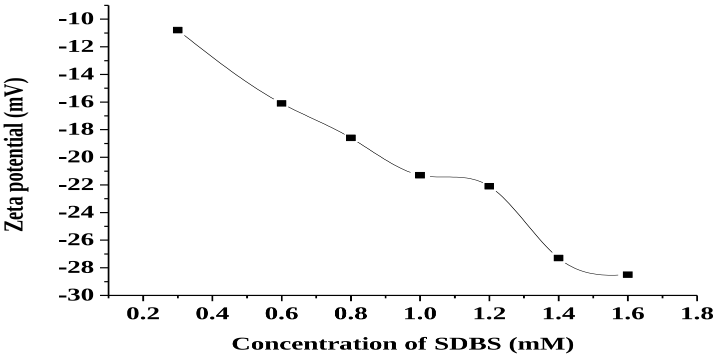


Figure 13. Variation zeta potential with respect to SDBS concentration.

Chapter 5:

Conclusion:

From the results obtained we have, in aqueous medium as weight percentage of corn starch increases viscosity increases and in case of cationic surfactant (Cetyl trimethylammonium bromide) as the concentration of surfactant increases viscosity increases gradually and reaches maximum there after decreases. In case of anionic surfactant (SDBS) as the concentration of surfactant increases initially viscosity decreases slowly there after decreases gradually. In aqueous medium zeta potential of corn starch is -8.94 mV which implies surface of corn starch in aqueous medium is negatively charged. As the concentration of CTAB increases Zeta potential value of starch particle increases, in case of SDBS surfactant medium as the concentration of SDBS increases zeta potential value starch particle decreases. Anionic surfactants are more effective in reducing the viscosity of corn starch slurry.

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