Rheological behaviour of hydrophobic mineral slurries (Coal Water Slurry)

Thesis Submitted in the partial fulfillment of requirements for the award of the degree of

DOCTOR OF PHILOSOPHY (Ph.D.)

in

METALLURGICAL AND MATERIALS ENGINEERING

By

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June - 2019

Dedicated

to

- **My beloved well wishers.**

DECLARATION

I hereby declare that the work described in this thesis, entitled "**Rheological behaviour of hydrophobic mineral slurries (Coal Water Slurry)**" which is submitted by me in partial fulfillment for the award of Doctor of Philosophy (Ph.D) in the Department of Metallurgical and Materials Engineering, National Institute of Technology, Warangal (Telangana.) – 506021, is the result of investigation carried out by me under the guidance of Dr. N. Narasaiah, Professor, National Institute of Technology, Warangal and Dr. S.Subba Rao, Chief Scientist (Rtd), CSIR-National Metallurgical Laboratory Madras Centre, Chennai-600113. The work is original and has not been submitted for the award of any Degree /Diploma of this or any other university.

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CERTIFICATE

This is to certify that the thesis entitled "**Rheological behaviour of hydrophobic mineral slurries (Coal Water Slurry)**" that is being submitted by Mr. M. Ananda Rao in partial fulfillment for the award of Ph.D. in the Department of Metallurgical and Materials Engineering, National Institute of Technology, Warangal is a record of bonafide work carried out by him under our guidance and supervision. The results embodied in this thesis have not been submitted to any other Universities or Institutes for the award of any degree or diploma.

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ACKNOWLEDGEMENTS

I would like to express my sincere gratitude and whole hearted thanks to my supervisor and philosopher **Dr. N.Narasaiah**, Professor, Metallurgical and Materials Engineering Department, National Institute of Technology, Warangal for his close association, invaluable guidance and encouragement at each and every stage of this research work. I would like to express my sincere gratitude and indebtedness to my co-supervisor **Dr. S. Subba Rao**, Chief Scientist (Rtd), CSIR-National Metallurgical Laboratory Madras Centre, for his close association, constant support, invaluable guidance, planning and execution of this work.

I am extremely grateful to **Dr. M.V. Pavan Kumar**, Assistant Professor, Chemical Engineering Department, National Institute of Technology, Calicut for his kind help, encouragement and valuable suggestions for successful completion of this research work.

I wish to sincerely thank our **Director, CSIR-National Metallurgical Laboratory Jamshedpur** and **Director, National Institute of Technology Warangal** for giving me an opportunity to carry out research work. I wish to express my sincere and whole hearted thanks to **Prof. M.K. Mohan (Rtd)** and **Dr. T.V.Vijaya Kumar, Scientist-In-Charge and** Mr **A. Ramesh, Asst Section Officer of CSIR-National Metallurgical Laboratory Madras Centre** for their valuable advice, generous encouragement in carrying this work.

I wish to express my sincere thanks to **Dr. C. Vanitha** (Associate Professor & HOD, DSC chairman ,MMED), **Dr.V.Vasu**, (Associate Professor, DSC member ,MED), **Dr.Asit Kumar Khanra** (Associate Professor, DSC member, MMED) and **Dr. G. Brahma Raju** (Assistant Professor, DSC member, MMED) for their support, generous encouragement in carrying out the project work.. I am also thankful to all other teachers, colleagues, friends who extended their help and moral support directly or indirectly during the research work. I thank all my family members who helped me directly or indirectly in achieving the goal.

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ABTRACT

Coal remains the largest fossil fuel resource in commercial energy generation due to industrialization and urbanization in India. Due to the non-availability of high rank coal reserves, the successful pre-processing and economic transportation of the coal for further and efficient utilization should be seen as an important step. The major requirement in preparation of coal water slurry (CWS) is that it should have higher coal concentration with minimum viscosity to allow ease of handling during the preparation, storage, and transportation. The extant literature over the subject reveals that the identification or synthesis of a suitable chemical dispersant is an influential factor for achieving favourable rheological characteristics.

Chemical additives namely Carboxymethylcellulose (CMC, Chemical Formula: $C_8H_{15}NaO_8$) and Sodiumtripolyphosphate (STPP, Chemical Formula: $Na_5P_3O_{10}$) have been proven as suitable dispersants for the preparation of the CWS and selected for the rheological studies of an Indian coal variety (*Coal 1* and *Coal 2*) mined in Jharkhand state. The proximate and ultimate analysis is used to characterise the coal, and zetapotential and turbidity measurements are carried to check the suitability of dispersant for preparation of CWS. The rheological properties of CWS are reported for different solid loadings (10%, 20%, 30%, 40% and 50%), dispersant dosages (i.e., CMC (0.5, 1.0, 1.5 and 2.0 kg/ton) and STTP (2, 4, 6 and 8 kg/ton)) and pH (4, 7, 8, 9, 10 and 12) at shear rates in the range of 60-160 s^{-1}. The effect of solids concentration, dispersant dosage and pH effects on CWS are comprehensively studied.

The rheological behaviour of CWS is investigated and compared for two different Indian coals (*Coal 1* and *Coal 2*) with respect to solids loading, dispersant addition, at constant pH-8 in the shear rate range of 60-160 s^{-1}. The dispersant addition is much effective for *Coal 1* in comparison to *Coal 2*. For a given dispersant, percent solids, a lower magnitude of shear stress versus shear rate, a wider distribution of flow behaviour index and favourable slurry pumpable characteristics are seen for *Coal 1* in comparison to *Coal 2* owing to their chemical nature and amount of ash-bearing mineral constituents present.

The rheological behaviour of the CWS prepared by *Coal 1* is investigated for two different dispersants namely CMC (polymeric) and STPP (non-polymeric) and compared for the effectiveness of dispersant addition. For a given solids concentration, a lower values of shear stress is reported for CMC as a dispersant in comparison to STPP. For a given solids concentration, the dispersant dosage required to attain per unit shear stress is lower for CMC in comparison to STPP. The coal surface with more negative surface charge resulted by the combination of steric effects and electrostatic repulsion is a prime reason for the same.

The rheological data is obtained in the shear rate range of 60-160 s^{-1} is successfully fitted for the power law model and flow behaviour index of each slurry was calculated. At lower solid loadings (10% 20%), the shear stress-shear rate relation do not alter with the dispersant dosage or pH and the slurries of exhibited dilatant behaviour. The slurry with 30% solid loading showed a transition from shear thickening to shear thinning behaviour with the increase in dispersant dosage and pH. For the higher solid loadings, the slurry exhibited shear thinning or pseudoplastic behaviour at higher pH values with the addition of dispersant. Interestingly, the transition from shear thickening to shear thinning nature was observed between 20% and 30% solids loading.

CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

Abbreviation	Description
SEM	Scanning Electron Microscopy
EDS	Energy Dispersive Spectroscopy
XRD	X ray diffraction analysis
CWS	Coal Water Slurry
STPP	Sodiumtripolyphosphate
CMC	Carboxymethylcellulose
ASTM	American Society for Testing and Materials
ADB	Air Dry Basis
HGI	Hardgrove Grindability Index
HCl	Hydrochloric acid
NaOH	Sodium Hydroxide
K	flow consistency index
n	flow behavior index
kg/ton	Kilogram per ton
Pa	Pascal
s^{-1}	Per second

CHAPTER-1

CHAPTER-1

INTRODUCTION

1.0 Introduction

Coal remains the largest fossil fuel resource in commercial energy generation due to industrialization and urbanization in India. The increased demand and use of maximum energy attracted the greater importance to address environmental issues. The idea of utilizing abundant and minable coal reserves against the backdrop of foreseen depletion of fossil-oil reserves has fuelled research on coal-based energy systems. Prior to utilization, these coals require cleaning and beneficiation process for upgrading the raw coal which is produced from the mining process. The investigations on the rheological behaviour of coal-water suspensions (CWS) have attracted great attention as slurry transport is an easy way of coal handling [1-3].

In recent years, direct combustion of a coal-water mixture of high energy density was also demonstrated. For this reason, the preparation of low viscous, high solids concentration CWS is desired for the transportation, beneficiation, and combustion. Coal-water slurry (CWS) is a scientifically proven technology for transportation and has been receiving intensive research since 1980 [4] and found excellent substitution as a fuel oil for diesel engines and gas turbine [5]. A major portion of Indian coal reserves are non-coking coal and low-rank coals. These low rank coals are used as raw feed material for gasification or combustion in the form of coal water slurry (CWS)[6].

As the excavated coal appears in different grades or varieties, nonetheless the rheological properties of CWS significantly depend on the quality and constituents of the coal, solids concentration, particle size distribution, functional groups, oxygen and moisture content, composition and hydrophobicity etc. The flow characteristics of the coal-water suspensions depends on (1) physical and chemical properties of the coal such as ash content, the amount of inherent water, the degree of coal oxidation, and the quantity of surface active functional groups; (2) the volume fraction, ϕ, of the suspension; (3) the particle size range and its distribution,(4) interparticle interactions in the suspension and their effects of pH and the chemical additives etc.[7]. A higher mineral matter and oxygen contents in the carbonaceous solids result in greater hydrophilicity, leading to increased adsorption of water on the solid surface [8]. For low-rank coals of higher ash content, preparation of CWS of higher coal concentration with proper flowability is a challenging task due to the dominant presence of mineral matter [9]. The reduction of friction in a non-settling slurry for long distance pumping can be achieved by reducing the viscosity of the slurry with the addition of suitable chemical additives [10].

Chemical additives are important ingredients in reducing the viscosity, maintaining fluidity and improving the stability characteristics of CWS. The chemical agents can introduce electrostatic or steric repulsions or increase the steric wettability of coal. The desirable characteristics of chemical dispersants were well narrated in Mosa et.al [11]. Much of the investigations were carried out in use of different dispersants for CWS like anionic, non-ionic and natural dispersants etc [12,13]. and polymeric dispersants, in particular, have been found to be effective additives in stabilizing the coal-water slurries [14]. Mishra and Kanungo [15] discussed, in detail, the influence of various physical and chemical factors on the flow characteristics of highly concentrated CWS. Addition of chemical dispersants is a widely practiced industrial method for the attribution of favourable flow characteristics to CWS. Tiwari et al [16] developed two anionic additives (1. naphthalene based, 2. naphthalene-toluene based) and tested them for the formation of stable and low viscous suspensions with two different coals. For a coal variety, successful slurry preparation beyond a certain limit of solid loadings was not possible due to the presence of more ash and oxygen-containing functional groups. Dincer et al [17] identified a suitable additive for the preparation of CWS with bituminous coal of Turkish origin. Kakuyi and Kamiya [18] developed anionic polymer dispersants for CWS preparation.

Pawlik [19] studied the effect of several low molecular weight polymers (non-ionic and anionic) as dispersants on the rheological properties of the CWS. The polyelectrolytes were found to be suitable as dispersants over the non-ionic polymers. Guo et al [20] investigated the effect of ultrasound irradiation on the rheological properties of CWS with a naphthene oil derived additive. The ultrasonic irradiation was found to increase the saturated adsorption amount value of the additive in the coal. A sulfonated acetone-formaldehyde resin [21-22], wheat straw alkali lignin resin [23], modified natural products [24-25], a mixture of surfactants [12] and polysulfonated condensates [26] were also successfully tested as dispersants for the CWS. Recently, Zhang et al [27] successfully synthesized and tested a novel humic acid-based polycarboxylic-type (HAP) dispersant. Earlier, carboxymethylcellulose (CMC) was used as an additive to impute favourable flowable characteristics to CWS [28-29]. Sodium tripolyphosphate (STPP) can be used as a dispersant in reducing the viscosity of coal-water slurries [11,29]. The dispersants which are amenable to the type of coal were added in small quantities to achieve lower viscosity and attribute stability to the slurry.

The major focus of investigations have mostly been on shear stress and shear rate relationship of slurries and flow behaviour to meet certain requirements such as ease of transportation and handling

1.1 Objective

For the slurry to be pumpable, in general, the viscosity must be as low as possible. The major requirement to prepare of CWS is that it should have higher coal concentration with minimum viscosity to allow ease of handling during preparation, storage and transportation. Chemical additives are important ingredients in reducing the viscosity, maintaining fluidity and improving the stability of CWS by introducing the electrostatic or steric repulsions or increasing the steric wettability of coal. The extant literature over the subject reveals that the identification or synthesis of a suitable chemical dispersant is an influential factor for achieving favourable rheological characteristics.

In the present investigation, carboxymethylcellulose (CMC) and sodiumtripolyphosphate (STPP), which have been proven to be suitable for the preparation of the slurry and were selected for the rheological studies of Indian coal varieties mined in Jharkhand state. The effects of different solids loading, dispersant dosages and slurry pH on flow behaviour were studied at a shear rate in the range of 60-160 s^{-1}. The rheology data was fitted for the power-law model and the flow behaviour index values were estimated to identify the rheological nature as a function of solids loading, dispersant dosage and pH of each slurry. The rheological properties of CWS were compared for the two Indian coal varieties in the presence of CMC and STPP as a dispersant at a pH value of 8. The effectiveness of the dispersant for the type of CWS was established with respect to rheological characteristics. The novelty of the work is the preparation of coal water slurries with low rank coals using easily available dispersants. The detailed rheological characterisation of each slurry of defined solid concentration, pH and dispersant loading is also a major contribution in this thesis work.

CHAPTER-2

CHAPTER-2

LITERATURE REVIEW

2.0 Rheology

Rheology is a study of plastic flow response of matter, under an applied force. The flow matter is generally in the liquid, soft solid or solid state. The rheological properties of particle suspensions can play major role in many industrial applications such as designing the pump for pipeline transportation of slurries etc. The data generated from the rheology can be used to find the relationship between flow rate and pressure drop. The rheological parameters can also be used to find the energy required to agitate the slurry in the tank, and for estimating the wear rate of the pipeline and its life.

The simplest model available to explain the rheological properties is called parallel plate model. Fig. 1 show the illustration of the model. The surface area of the top plate is “A”, and is moved by a force “F” at a speed of “v.” The bottom plate remains at static condition. The distance between the plates that the flow of materials under consideration, is given by “h”. The thinnest elements of the liquid will be displaced between the plates.

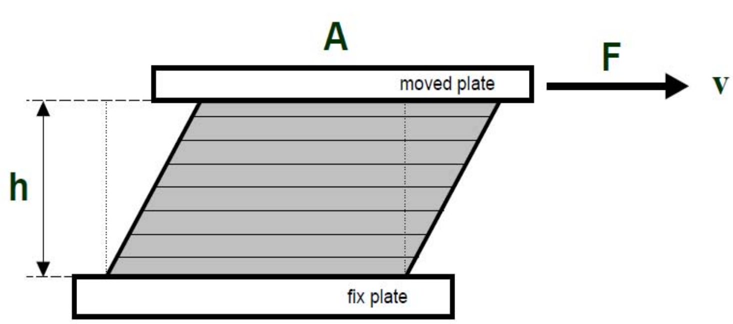


Figure 1. Schematic diagram of parallel plate model

The **shear stress** is the Force (F) acting on unit area (A) to displace the liquid element between the two plates. The shear stress caused by frictional forces between fluid particles due to fluid is given by "**τ** ", and is denoted as N/m^2 or Pa

Shear stress (τ) = Force (F) / Area(A)

The **shear rate** is rate of change of velocity at which one layer of fluid passes over an adjacent another layer. The application of shear stress on the fluid generates the laminar shear flow between the two plates and velocity differential. The layer on the uppermost side moves at the maximum velocity Vmax, while the layer at lowermost side remains at static. Then the shear rate can be denoted as "γ " and expressed as

Shear rate (γ)= dv/dh

Where, dv= velocity differential of flow layers

dh= thickness differential of the flow layers

2.1 Rheological properties of a fluid

The type of flow behaviour of the fluid depends on the solid concentration of the suspension and its viscosity. Fluids can be broadly classified as Newtonian and Non-Newtonian type.

2.1.1 Newtonian fluids

The fluids which follow the constant viscosity with strain rate or the shear stress directly proportional to strain rate are called Newtonian fluids. Newtonian fluids always follow the Newton's law of viscosity. Fig.2(a-b) show the graphical representation of Newtonian fluids

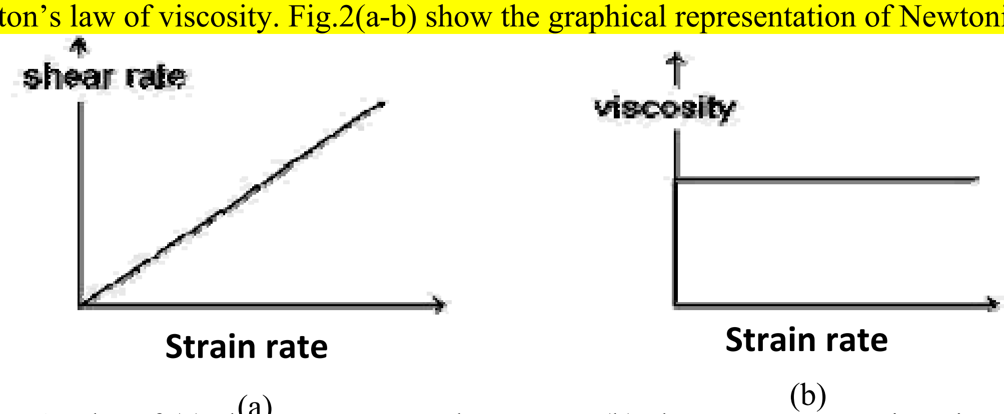


Figure 2. Plot of (a) shear rate versus shear stress (b) shear rate versus viscosity for Newtonian fluids.

2.1.2 Non-Newtonian fluids

The Non-Newtonian fluids do not follow the Newton's law of viscosity. The solids concentration, the particle size, shape and their distribution determines the nature of Non-Newtonian behaviour in case of slurries. The Non-Newtonian fluids can be classified as pseudoplastic fluids and dilatant fluids.

2.1.2.1 Pseudoplastic fluids

The pseudoplastic fluids are type of fluids whose viscosity decreases with the increase in shear rate (Fig.3 (a-b)). These fluids can also be called as shear thinning fluids. The mathematical expression for pseudo plastic fluids according to "Ostwald de Waele" in equation (1)

$$\tau = K \cdot \gamma^{n} \qquad (1)$$

Where n < 1 for pseudoplastic materials

Examples: Suspensions, Paints, Dispersions, Lotions, Gels, Creams.

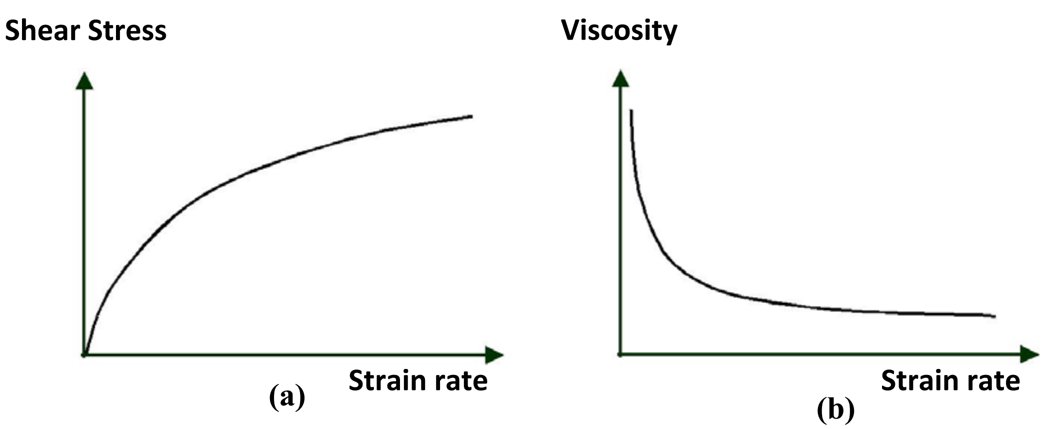


Figure 3. Plot of (a) flow curve of pseudo plastic and (b) viscosity curve of pseudo plastic materials

2.1.2.2 Dilatant fluids

The fluids whose viscosity increases with an increase in shear rate are called as dilatant fluids. These fluids can also be called as shear thickening fluids (Fig.4(a-b)). The mathematical representation of dilatant fluids according to "Ostwald de Waele" in equation (1) (as shown in Chapter 2, Section 2.1.2.1)

$$\tau = K . \gamma^{n} \qquad \text{............................} \qquad (1)$$

where n > 1 for dilatant materials

Examples: Wet sand, Concentrated corn starch, Ceramic suspensions, Surfactant solutions.

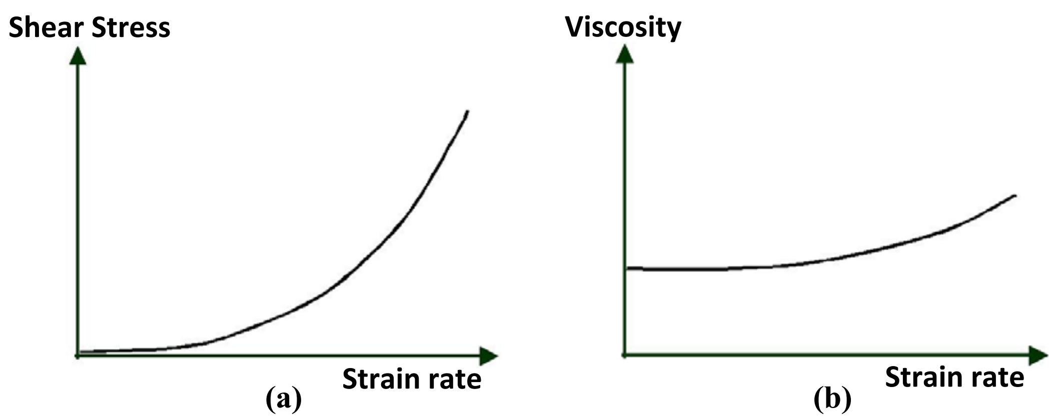


Figure 4. Plot of (a) flow curve of dilatant material and (b) viscosity curve of dilatant material

2.3 Rheological Measurements

Rheometer is a laboratory device used to find the slurry behaviour when forces are applied. A rheometer which controls either the applied shear stress or shear strain is called rotational or shear rheometer. Instrument used which control a user-defined shear strain and measure the resulting shear stress is called native strain controlled instrument. For user-defined shear stress to measure the resulting shear strain is called native stress-controlled instrument. When the annulus is filled with liquid and the cylinder is rotated at defined speed against the liquid, the resulting drag force on the cylinder is measured as torque and can be converted to a shear stress.

2.4 Coal Formation & Types of Coal

Coal is formed by the combined effects of biological, physical and chemical processes on ancient shallow swamps of plant deposits under the action of temperature and pressure for over millions of years. The interrupted process of decaying and preventing the release of the stored solar energy decides the degree of alteration (metamorphism) and determines the rank of the coal. The relative amount of moisture, volatile matter and fixed carbon content in the position of coal in the coalification series i.e., from peat, lignite, bituminous and anthracite. Generally, a lower amount of moisture and volatile matter, and higher carbon indicates the enhanced rank of the coal (carbon content is low in peat and high in anthracite).

(i) Peat is the first sediment formed in the coalification process and appears as moist and spongy material. Practically peat cannot be considered as coal but can be used as source of energy in some of the applications. The amount of water contain in the peat will be 90%.

(ii) Lignite is considered as the lowest rank of coal and its heat value varies in between 4,000 to 8,300 British Thermal Units (BTU) per pound. Lignite contains 60–70% carbon content and is crumbly in nature. Lignite can be used the place where the efficient fuel is not available.

(iii) Sub-bituminous coal is a low rank coal and its properties falls in between lignite and bituminous coal. The heat value of the sub-bituminous coal lies between 8,300 to 11,500 BTU per pound. Sub bituminous coals contain 70 to 76% carbon and are relatively low in density and high-water content.

(iv) Bituminous coal is a tar-like, soft, smooth and tiny layered. The rank of the bituminous coal is higher in comparison to lignite but poor in quality in comparison to anthracite. The heat value of bituminous coal is in between 11,500 to 15,500 BTU per pound. The carbon content of bituminous coal is around 60–80%. Bituminous coals find application in steel and iron industries as a source of energy.

(v) Anthracite is a hard, compact, deep black, glassy and highest rank of coal. The heat value of the anthracite is around 15,000 BTU per pound and is considered as a big energy producer. These coals contain 92 to 98 % of carbon and possess highest energy density.

(vi) Graphite is generally considered as a highest rank of coal in coalification series. Graphite cannot be used as fuel and can find application in manufacture of pencils, in self lubrication and dry lubrication etc.

2.5 Coal Water Slurry (CWS)

In countries like India where high-quality coal is scarce, the successful pre-processing and economic transportation of the coal for further and efficient utilization should be seen as an important step. A large amount of pulverized wet coal is produced in the mechanized coal mining processes worldwide. The extracted coal need to be stored, handled and transported for subsequent preparation or beneficiation processes like pelletization, washing and flotation etc. The transportation of coal as CWS is an effective transportation method as it can be carried out through pipes with minimum cost and energy consumption. Using low-rank coals having higher ash content, preparation of CWS of higher coal concentration with proper flowability is a challenging task due to the dominant presence of mineral matter. A good understanding of the rheological properties of coal-water slurries is essential for design and optimization of the processes.

Utilization of high ash or low-rank coals as a liquid fuel in the form of CWS is a big challenge. An initial attempt to utilize low-rank coals in preparation of CWS consisted of simply mixing the pulverized low-rank coals in its natural state directly with water. The net result of the utilization of low-rank coals in its natural state was not economically feasible due to its extremely low energy content and unfavourable characteristics before and after burning [30]. However, the introduction of chemical additives has made the utilization of low-rank coals utilization as CWSs possible and feasible [31].

Ideally, the CWS with maximum coal loading should exhibit good rheological behaviour and relatively stable at a static state and during transportation. The most important input data needed for the design of the slurry transportation system is the rheological behaviour of the slurry at various concentrations and flow conditions. The data is used to find the flow rate-pressure drop relationship during transportation. Moreover, knowledge of rheological behaviour of CWS can lead to the enhanced ability to control flow behaviour and can be exploited by the different coal beneficiation unit operations.

2.5.1 Coal Water Slurry parameters

Parameters such as particle size and distribution, the mass fraction of fine particles and solid concentration in slurry etc. will play a major role in deciding the characteristics of the slurry.

i. Particle size and distribution: The relative amount of mass of the particles present according to size in the slurry can be considered as particle size distribution. The particle size distribution can be determined by passing the solids through different screens of varying mesh sizes.

ii. Mass fraction of fine particles**:** The particles of size less than 75mm can be considered as mass fraction of fine particles. The ideal percentage of small particles in the slurry must be atleast 50% by weight.

iii. The concentration of solids: The amount of solids present in the total volume of the slurry can be defined as concentration of solids. Generally, the concentration of solids in slurry can be measured by the volume or weight of the slurry.

2.6 Effect of Additives/Dispersants on Slurry

The dispersability of slurries depends on parameters such storage time, particle size, solids concentration, pH and type of dispersant its dosage [28]. An additive/dispersant will be added to a slurry to improve the separation of particles by inducing the surface charge on the particle and to prevent settling of the particle. Dispersants/additives will deflocculate the solid particles and thus significantly reduce the viscosity of slurry. This facilitates the preparation of slurry with a maximum solid concentration. The dispersant molecules adsorb on the particle surface and induce repulsion among the particles. Basically, two mechanisms will contribute to the dispersion namely electrostatic stabilization and steric stabilization.

The process of repulsion by the particles carrying a charge of same sign is called electrostatic stabilization. The process of particles covered with tails dissolving in the liquid and surrounding the particles is called steric stabilization. The repulsion will result from both the mechanisms that may either be applied separately or in combination. When particles are dispersed in a medium, they form at random chains by Vander Waals' forces or bonds. The attractive forces predominate over a range of inter particle distances in the case of repulsive forces. Positive charge surfaces will be created in the case of attractive forces present.

In general, when the dispersant is added to the slurry, the interaction between the particles will be reduced by the negative attraction and thus lowers the viscosity of the slurry. Hence the dispersant addition facilitates the slurry for better flow properties.

2.7 Particle Surface Charge & Zetapotential

The surface chemistry plays major role in developing interfacial reactions between the solid-liquid and solid-gas [32]. The surface charge created on the particle surface can be defined as the electrical potential difference between the inner and outer surface of the dispersed phase. The developed surface charge on the particle surface greatly depends on the pH of the liquid phase due to the generation of H^+ and OH^- ions.

The stability of the colloidal dispersions will be indicated by the zetapotential [33-34]. The potential difference between the dispersing medium and the stationary layer of liquid attached to the dispersed particle is called zetapotential.

2.8 Literature review on Coal Water Slurry

A review on the previous published literature laid foundation and formed basis for the work in present investigation. A better understanding about the definition of the research problem of the thesis was arrived after the review of scientific literature on the CWS preparation and their reported rheological characteristics.

A.R. Hasan et al. [35] studied the rheological behaviour of low rank coal water slurries. A Sarpy Creek Sub-Bituminous coal from Montana was used for the investigation. The rheological behaviour of the slurry made of the as received coal was compared with the slurry made from hot water dried coal. Both the coals exhibited the pseudo plastic behaviour and lower viscosity values were observed for the slurry made of hot water dried coal.

Roh N.S et al. [36] investigated the rheological behaviour and stability of the coal water slurry and discussed the effect of coal type, coal concentration, coal particle size distribution and stability. Seven bituminous coals were used for the studies, which are originated from Grace, Getty, South African, Australian, American, Tatung 1 and Tatung 2. Formaldehyde condensate of sodium naphthalene sulphonate (anionic type) was used as dispersant for the studies. The results showed more viscous nature for coal water mixture as the mean particle size decrease, but less viscous with decrease in equilibrium moisture content of the coal and solids volume fraction. The mixing of coarse and fine particles was found to be very effective in obtaining mixtures characterised by high solids content and low viscosity. Coal water mixture viscosity was lowest when the blending ratio of fines was ~35 wt.%, irrespective of mean size ratio. The particle size distribution which gives high fluidity was appeared to enhance the stability of suspension.

Nguyen et al. [37] studied the rheological behaviour of the coal water slurries as a function of solids concentration, particle size and size distribution. Two low rank coal deposits of Lochiel and Bowman's from South Australia were studied in the present investigation. The coal water slurries were prepared by dispersing the coal particles finer than 45 microns in water. Coal water slurries exhibited wide spectrum of flow behaviour ranging from Newtonian at lower solids concentration to shear thinning and viscoplastic at higher solids concentration. The investigation highlights the possibility in preparation of optimum coal water slurry containing low viscosity at higher solids concentration by controlling particle size distribution.

G. Atesok et al. [38] investigated the effect of coal properties on the viscosity of coal water slurry. Three different ranks of coals originated from Siberia, Soma and Istambul-Agacli of Turkey are used for studies. Sodiumpolystyrenesulphonate (PSS) and sodium salt of carboxymethylcellulose are used as dispersant and stabilizer respectively. The effect of zetapotential, viscosity on rank of the coal was investigated. Zetapotential found to be decreased dramatically with addition of chemical additive. The adsorption density of PSS decreases in the order of decreasing coal rank. Low rank Turkish coals used for this investigation permit less solids loading capacity compared to the Siberian coal for the same slurry viscosity.

Mishra et al. [39] studied the effect of solid concentration, ash content, pH and temperature on rheological behaviour of coal water slurry. The coal originated from Talcher coal field, Orissa, India, was separated into three categories by hand picking, jigged ground ROM (Run- of -mine), ground ROM. The investigation concluded that the slurry became more viscous with increase in ash content and solid concentration. The apparent viscosity was found to be highest at pH 6 and lowest around pH 8 for all three categories of coal samples. The CWS under investigation shows the Non-Newtonian flow behaviour at lower pH. The research highlights the independent nature of relation between apparent activation energy, shear rate and solid concentration.

H. Dincer et al. [17] investigated the effect of different chemicals that were used as dispersant and stabilizer on the stability and viscosity of CWS. Coal water slurries were prepared by using Bituminous coal sample from Zonguldak region of Turkey. Derivative of carboxylic acid, napthalenesulfonate-formaldehyde condensate and polyisoprenesulphonic acid soda were used as dispersants and sodium salt of carboxymethylcellulose (CMC-Na) was used as stabilizer respectively. The results show that the anionic dispersing agents of polyisoprenesulphonic acid soda type were more effective in decreasing viscosity and increase in stability. And also established, the use of polymeric dispersing agents would be more economical in allowing maximum solids concentration in preparation slurry.

Kaushal. K. Tiwari et al. [16] studied the rheological studies of highly concentrated coal water slurry using Ledo coal of Makem field in Assam and Sirka coal of north Kanranpura field, Jharkhand India. The effect of dispersant on coal solids concentration, dispersant addition was established. The coal solids concentration was varied from 56 wt.% to 70 wt.%. Two anionic chemical additives namely naphthalene- based and naphthalene-toluene-based additives were used in preparation of slurry. For a given solids concentration between 65-70 wt.%, the addition of naphthalene- based and naphthalene-toluene-based are much effective in reducing the viscosity of the slurry for dosage concentration of 0.8 and 0.9 wt. % respectively.

Boylu et al. [40] studied the effect of coal particle size distribution, volume fraction and rank on the rheology of coal water slurry. Investigation was carried out by using two Turkish lignites from Soma and Istanbul – Agacli, and a bituminous coal from Siberia. The coals of different ranks exhibit different chemical and physical properties such as porosity, specific surface area oxygen/ carbon ratio, etc. The viscosity decreased from lower rank to higher rank coals. The coal water slurry prepared using higher rank coals could contain higher amount of solids. The viscosity increased with increase in pulp density by weight for various particle size distribution.

F. Boylu et al. [28] studied the effect of carboxymethylcellulose (CMC) on the stability of coal-water slurries using two coals from Soma and Istanbul, and coal from Zonguldak. This study emphasizes the importance of the level of inorganic material in coals in the stabilization of coal-water mixtures. And also demonstrated the stability property of anionic CMC in coal water mixtures prepared from both Turkish bituminous and lignite coals that have hydrophobic and hydrophilic surfaces. The polymeric structure of CMC and anionic properties due to its carboxylic groups, does not have an important effect on coal-water mixtures prepared from lignite coals that have hydrophilic surfaces. The inorganic material with hydrophobic surface acts as a stabilizer in CWS and prevents sedimentation.

Marek Pawlik et al. [19] investigated the effect of several low molecular weight polymers (MW<100000) on the surface properties of a medium volatile bituminous coal in concentrated aqueous suspensions through adsorption, flotation, electroacoustic and rheological measurements. The medium volatile bituminous coal from Fording mine (British Columbia

Canada, used for preparation of slurry. The anionic polymers namely carboxymethylcellulose, polystyrene sulphonate and humic acids are used as chemical additives for preparation of slurry. They identified the dispersant capabilities of the polymers not only depend on their ionic / non-ionic character but also on the ability to increase the wettability of coal surface. The anionic polymers are much stronger coal dispersants since their action is a combination of steric and electrostatic repulsive forces. In contrast the non-ionic polymers can only act through steric effects. Hence demonstrated that, the rendering the coal particles hydrophilic is the common mode of dispersing action of both anionic and non-ionic polymers.

Eisa S. Mosa et al. [11] Investigated the effect of chemical additives on flow characteristics of coal water slurry. The coal originated from El-Maghara coal mine, Northern Sinai, Egypt was used for the studies. The power law model is used to characterize the flow behaviour of CWS. Sulphonic acid, sodiumtripolyphosphate and sodiumcarbonate were used as dispersants, and sodium salt of carboxymethylcellulose, Xanthan gum were used as stabilizers in preparation of coal water slurries. Among these dispersants sulphonic acid found to be better reducing the viscosity. Among stabilizers sodium salt of CMC was found to better in comparison to Xanthan gum.

S. K. Mishra et al. [4] discussed the importance of factors effecting the preparation of highly concentrated coal water slurry. He discussed the effect of surface properties of coal in different aqueous medium, effect of inorganic mineral matter in the coal, effect of coal macerals, the effect of oxygen containing functional group, porosity, water content and particle size distribution. And he also discussed the importance of surface-active agents in preparation of highly concentrated coal water slurry.

Wei Yuchi et al. [41] studied the effect of coal characteristics on the properties of CWS using sixteen Chinese coals of different ranks from Lignite to Anthracite. In this study the slurriability, rheological behaviour and static stability are examined for coal rank, air equilibrium moisture, maximum moisture holding capacity, ash content, surface properties, petrographic macerals, pore structure and adsorption characteristics of dispersant. The content of soluble ions showed a positive effect on the static stability of CWS.

Mingsong Zhou et al. [42] investigated the studies on adsorption and the zeta potential on coal water interface. The effect of the molecular weights of sodiumlignosulfonate on the apparent viscosities of CWS was studied. The results show that adsorption behaviour of dispersant was the key factor in dispersant effect. The higher adsorption amount and compact adsorption film help to reduce the viscosity of CWS. Zetapotential influenced by sulfonic group and carboxy content of the lignosulfonate molecule.

Senapati et al. [43] investigated the rheological behaviour of coal water slurry using natural additive prepared from drupes. Two types of coals were obtained from Talcher Coal Field, Orissa, India which differ in ash contents. The coal water slurries were prepared with concentration by weight ranging between 55 - 63.7 %. The additive concentrations for coal water slurries were varied from 0.4–1.2 % by weight. The rod penetration test was used to measure the static stability. They found that the coal water slurry in the presence of natural additive exhibited Bingham plastic behaviour. The static stability of the coal water slurries was found to be 3 to 4 weeks by employing the natural additive.

D.Das et al. [44] prepared highly concentrated coal-water slurry from three different low-rank coals of Indian origin having variable ash content. Saponin extracted from the seeds and pericarps (mods) of the Acacia concinna plant were used as dispersants in investigating the rheology and stabilization of the slurry. The slurry was found to stable when the saponins extracted from both the seeds and pericarps of the plant. They claimed that the plant-based additive saponin from A. concinna (both pericarps and seeds) can be replaced for a synthetic additive, such as SDS (Sodium Dodecyl Sulphate).

Zhou et al. [45] studied the rheological properties of concentrated coal-water slurry and flow characterized by using Herschel-Buckley model. Four kinds Chinese coals representing Bituminous (Panjang) and Brown coal (Yangzhou, Datong, Shenhua) are used for preparation of CWS. Lignin-based dispersant (MSL) was used as an additive for the slurry. Coal concentration of 64.0 wt.% and dispersant dosage of 0.7 wt.% and 1.5 wt.% were used to prepare two slurries. They found that the slurry showed shear-thinning characteristic when the dispersant dosage was 0.7 wt.% and for 1.5 wt.% dosage and also it showed shear-thickening characteristics. The effects of factors such as solid content and dispersant dosage on rheological property of CWS were studied, and the results showed that with increasing coal concentration and tend to pseudoplastic characteristic whereas, with increasing dispersant dosage the slurry tend to dilatant flow characteristic.

Buranasrisak et al. [46] studied the rheological behaviour of coal water slurry based on characteristics like particle size distribution and packing. Samples were prepared from sub-

Bituminous coal of Indonesian origin. Naphthalene Sulfonate formaldehyde (NSF) was used as dispersing agent and Na-CMC was utilized as the stabilizer. The coal water slurries at different solid loadings ranging between 60 to 65 % by weight were tested. Monomodal, bimodal and multimodal distributions at different coarse to fine ratios were prepared with different packing characteristics of the coal samples. They observed that the coal water slurry made from bimodal particle size distribution shows maximum coal loading capability.

M.Zhou et al. [47] studied a new polycarboxylic acid (PC) hyper-dispersant containing fundamental chain, sulfonic, carboxyl, poly oxyethylene groups is designed and synthesized as additive to prepare the CWS with ideal solid content, viscosity and stability. The slurry exhibited the shear thinning characteristics, and found to be advantageous for static stability, pipe pumping and spray combustion under high shear condition. The excessive PC dosage weakens the pseudo plastic characteristics of CWS, thus the above dispersant is well suited for reduction of viscosity and improving stability.

Mani Kanwar Singh et al. [48] studied the rheological behaviour of CWS of using coal from Assam, India. The effect of particle size, solid concentration and temperature on rheology of coal water slurry has been investigated. The rheological behaviour of slurry was analysed by blending the coal samples with the mixtures of coarse and fine particles and hence making a bimodal particle size distribution. The slurry having the bimodal particle size distribution was prepared by blending the fine particles of 53-75µm with coarse particles of 106-150µm as well as with 150-250µm. They found that the bimodal slurry sample having 30% coarse particle at wide range of concentration possess the minimum viscosity and is preferable for slurry transportation. They suggested Herschel-Buckley model was a suitable model for flow characterization of slurry concentration more than 30%.

D. Das et al. [22] investigated the stability of concentrated coal water slurry using mixture of natural and synthetic surfactants. Saponin as natural surfactants and Hexadecyltrimethyl ammoniumbromide (cationic), sodiumdodecylsulphate (anionic) are used as synthetic surfactants for the stability studies. Three different low rank coals from Talcher, Orissa, India were used for present studies. The mixture of saponin and sodium dodecyl sulphate found to be effective for stabilizing coal water slurry.

Brian.P. Williams et al. [49] studied the rheological properties of petroleum coke water slurries using variety of non-ionic and anionic dispersants. They petroleum coke obtained from S

K innovation in Korea. They studied the effect of pet coke loading and dispersant on yield stress, surface coverage and adsorption and effect of Xanthan gum on stability of CWS. Pluronic F127 is found to be better dispersant for making stable CWS.

Amrita Mukherjee et al. [50] studied the effect of hydrophobicity on viscosity of carboneous solid-water slurry. Three different coals were selected for slurry preparation based on the carbon content namely pet coke, bituminous coke, Illinois #6. The effect of chemical additives namely ammonium lignosulfonate, sodium polystyrene sulfonate and octylphenol ethoxylate are used for the studies. Octylphenol ethoxylate is found to be effective in reducing the viscosity of pet coke and bitumen water slurries. Ammonium lignosulfonate, sodium polystyrene sulfonate is found to be better additive for non-hydrophobic bituminous Illinois #6 coal.

Ahmet Gurses et al. [53] studied the effects of parameters such as coal loading, initial pH of mixture, the addition of various electrolyte, surfactants, temperature on the viscosity and rheological parameters of coal water mixture. The coal sample used in the study was from Askale-Erzurum region, Turkey. Studies were carried out using $AlCl_3$ and K_2HPO_4 as electrolytes and cetyltrimethylammoniumbromide, sodiumdodecylsulphate and borosperse NA-3A as surfactants. Cetyltrimethylammoniumbromide and K_2HPO_4 was found to be most effective additive in reducing the viscosity of CWS.

From these studies, it can be seen that rigorous rheological characterisation of coal water slurries, especially with lean or low rank coals was not reported in the literature. Hence, in this thesis work, the rheological characteristics of two types of Indian coals which are characterized as low rank coals were investigated. The salient parameters of CWS preparation are identified and these variables (solids loading, dispersant loading, pH). The method and procedure for the rheological characterisation can be used for further studies on the rheological behaviour of coal water slurries. This work is industrially relevant as the transportation of coal is an integral part of many thermal power plants, chemical and process industries.

CHAPTER-3

CHAPTER-3

EXPERIMENTAL DETAILS: MATERIALS & METHODS

3.1 Introduction

This chapter describes the materials and methods applied to accomplish the aims of this work. The coal analysis, scanning electron microscopy and x-ray diffraction are performed to suitably obtain the feed sample is outlined in section This is followed by Section, which describes the experimental methods applied to investigate the suitability of dispersant for CWS by zetapotential and turbidity techniques. Finally, experimental methods performed to rheological techniques are discussed.

3.2 Coal sample

The coal samples used for this study were originated from the Jamadoba (*Coal 1*) and North Karanpura (*Coal 2*) of Jharkhand state respectively. About 20 kg of coal sample was crushed (Insmart make Jaw crusher) and ground (Insmart make Roll crusher) to half an hour in ball mill at 70% solid - liquid ratio. Samples were air dried for a week and drawn 10 kg of sample by using standard sampling methods. The milled sample was separated into several size fractions by screening. The size analysis of the coal sample (*Coal 1*) used for the preparation of CWS and to study the rheological behaviour in the presence of dispersant is given in the Table 1. The comparative studies on flow behaviour of the CWS with respect to type of coal (*Coal 1* and *Coal 2*) and nature of dispersant used are carried out for the coal sample in the size range of -105 + 38 (mesh).

3.3 Proximate and Ultimate analysis

Proximate and ultimate analysis test was done on coal samples to characterize the different percentages of ash, total sulphur, volatile matter, inherent moisture content, carbon, hydrogen, oxygen and nitrogen as well as the calorific value of the coal. Tests methods were performed as per the ASTM (American Society for Testing and Materials) accredited standards.

Table 1. Size analysis of *Coal 1* sample

Mesh size	Cumulative weight % (Pass through)
+150	100
-150+105	98.49
-105+74	90.01
-74+53	78.29
-53+38	70.37
-38+25	58.82
-25+16	47.78
-16+11	39.58
-11+5	24.03
-5+4	20.47
-4	0
	D_{80}=56.23 microns

3.4 X-Ray Diffraction Analysis

The quality and mineral content in the coal is determined by X-ray diffraction method. The analysis of phase constitution was carried out using Bruker D8 Advance X-ray diffractometer (XRD). The samples were analysed for a 2θ range of 10° to 80° with a step size and scan time per step of 0.02° and 5 s, respectively. Cu-Kα radiation with Ni filter was used for the present measurements. The quantitative phase analysis was performed using MAUD (Material Analysis Using Diffraction) software. The most common occurring minerals in the coal are quartz, kaolinite, muscovite, pyrite, carbonates – calcite, dolomite, siderite and oxides – magnetite, hematite.

3.5 Scanning Electron Microscopy (SEM) and Energy dispersive spectroscopy (EDS)

The morphology of the coal sample (texture), chemical composition, and crystalline structure are determined using the scanning electron microscope (SEM) make-FEI-Nova Nano SEM. Magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm. Qualitative or semi-quantitative chemical compositions were performed using energy dispersive spectroscopy technique. Energy Dispersive Spectroscopy Analysis (EDS) was an x-ray technique used to identify the elemental composition of materials. The data generated by EDX analysis consist of spectra showing peaks corresponding to the elements making up the true composition of the sample being analysed. Elemental mapping of a sample was used for qualitative, semi-quantitative, quantitative and also to provide spatial distribution of elements (determination of the concentrations of the elements present).

3.6 Dispersants

Two anionic dispersants were used as dispersing agents for rheological studies, namely Carboxymethylcellulose (CMC, ChemicalFormula:$C_8H_{15}NaO_8$) and Sodiumtripolyphosphate (STPP, Chemical Formula: $Na_5P_3O_{10}$). CMC is an anionic polymeric dispersant and STPP is an anionic inorganic dispersant. Four distinct concentrations of the dispersants, i.e., CMC (0.5,1.0,1.5 and 2.0 kg/ton) and STTP (2, 4, 6 and 8 kg/ton) were tested. For the CWS of given solids loading and dispersant dosage, the pH (4,7, 8, 9, 10 and 12) also varied.

3.7 Zetapotential

The zeta potential measurements were investigated using Beckman Coulter Delsa™Nano C Particle Analyser. In all experiments, 0.2 g of -75 µm size coal sample (*Coal 1* and *Coal 2)* was conditioned for 5-10 minutes at room temperature in a 100 ml of solution. Four distinct concentrations of each dispersant namely carboxymethylcellulose (0.5, 1.0, 1.5 and 2.0 kg/ton) and sodiumtripolyphosphate (2.0, 4.0, 6.0 and 8.0 kg/ton), were added and prepared to 100 ml suspension. The prepared solution was made to undergo zetapotential tests. All the zetapotential measurements were carried out with an error percentage of less than ±5.

3.8 Turbidity

The turbidity measurements were carried out using Digital Nephelo Turbidity Meter 132. For this purpose, a 100 ml of well mixed suspension was prepared with one gram of coal (*Coal 1* and *Coal 2)* followed by 10 times dilution of the solution. The diluted solution was made to undergo the turbidity tests for different concentrations of the chemical dispersant, carboxymethylcellulose (0.5, 1.0, 1.5 and 2.0 kg/ton) and sodiumtripolyphosphate (2.0, 4.0, 6.0 and 8.0 kg/ton) respectively. All the turbidity experiments were carried out with an error percentage of less than ±5.

3.9 Rheology

The rheological properties of the slurries were tested by cup and bob Rheometer (Anton Paar physica 101 make) in the shear rate range of 60-160 s^{-1} using CC 39 sensor system. The temperature was kept constant at 24^0C by using proper cooling system. 15 data points were measured for each test. The rheological experimental setup is shown in Fig 5. Five different coal water slurries (*Coal 1* and *Coal 2)* of varying solid concentration (10%, 20%, 30%, 40% and 50%) were prepared for this purpose. Four distinct concentrations of the dispersant, i.e., STTP (2, 4, 6 and 8 kg/ton) and CMC (0.5, 1.0, 1.5 and 2.0 kg/ton) were tested. For the CWS of given solids loading and dispersant dosage, the pH (4, 7, 8, 9, 10 and 12) also varied. The schematic of detailed research plan is given in Fig.6

Distilled water was used in preparation of dispersant solution. Each slurry sample was thoroughly mixed with dispersant using a stirrer before conducting experiment. 60 ml slurry was used for each experiment. The amount of addition of coal, water and dispersant to make defined slurry concentration are calculated based on the standard procedure. All the rheological experiments were carried out with an error percentage of less than ±5.

To describe the flow behaviour of CWS (Newtonian or Non-Newtonian), flow behaviour index "*n"* of the power law model is employed in the rheological characterization. The power law model is given in equation-(1) (as shown in Chapter 2, Section 2.1.2.1)

$$\tau = K\dot{\gamma}^n \qquad (1)$$

where τ and $\dot{\gamma}$ are shear stress and shear rate or velocity gradient respectively. The values of flow consistency index (K) and flow behaviour index (n) are dependent on the nature of the fluid. For n=1, the fluid is Newtonian type. In the case of Non-Newtonian nature of the fluid ($n \neq 1$), the rheological nature of the fluid is denoted as dilatant (n>1) or pseudo-plastic (n<1) based on the n value. Rheoplus software was used for the estimation of flow behaviour index using power law model.

Figure 5. Experimental setup of Anton Paar Physica MCR101 Rheometer

3.10 Volume of water and dispersant calculations

Volume of water and dispersant calculated by the following standard procedure and is given in the equations (2)-(4)

$$\frac{C_w}{\rho_p} + \frac{100 - C_w}{\rho_w} = \frac{100}{\rho_{sl}} \qquad (2)$$

$$\text{\% Solids by Weight} = C_w = \frac{\text{Weight of the solids}}{\text{Weight of the pulp}} \times 100 \qquad (3)$$

$$\text{Pulp (Slurry) density} = \rho_{sl} = \frac{\text{Weight of the pulp}}{\text{Volume of the pulp}} \qquad (4)$$

Example: Calculation of weight of particles and weight of water required to maintaining the 30% solids (C_w=30%) in 60ml volume of the pulp.

Density of the coal sample ρ_p= 1.4gm/cc

Density of the water ρ_w= 1.0gm/cc

Density of pulp ρ_{sl}= 1.0937 gm/cc

Weight of the pulp =65.64 gm

By using above formula,

Weight of the solids=19.96 gm

$$\text{Density of solids} = \frac{\text{Weight of the solids}}{\text{Volume of the solids}}$$

Volume of solids=19.96/1.4 = 14.2ml

Total volume of slurry=Volume of solids+ volume of water

Volume of water without dispersant =60-14.2=45.8ml

If 2kg/ton dispersant is added for 19.96gm coal material then,

- 2 kg dispersant per 1000kg coal
- 0.002gm dispersant for 1gm
- For 19.69gm of coal required amount of dispersant is =19.96X0.002=0.03992gm

2% dispersant solution is prepared by dissolving 2gm in 100ml water.

0.03992gm of dispersant=(100/2)X0.03922=1.996ml dispersant

Volume of dispersant for 2kg/ton dosage at 30%solids=1.996ml

Water required =Actual volume of water - volume of dispersant

= 45.8 - 1.996

= 43.804ml

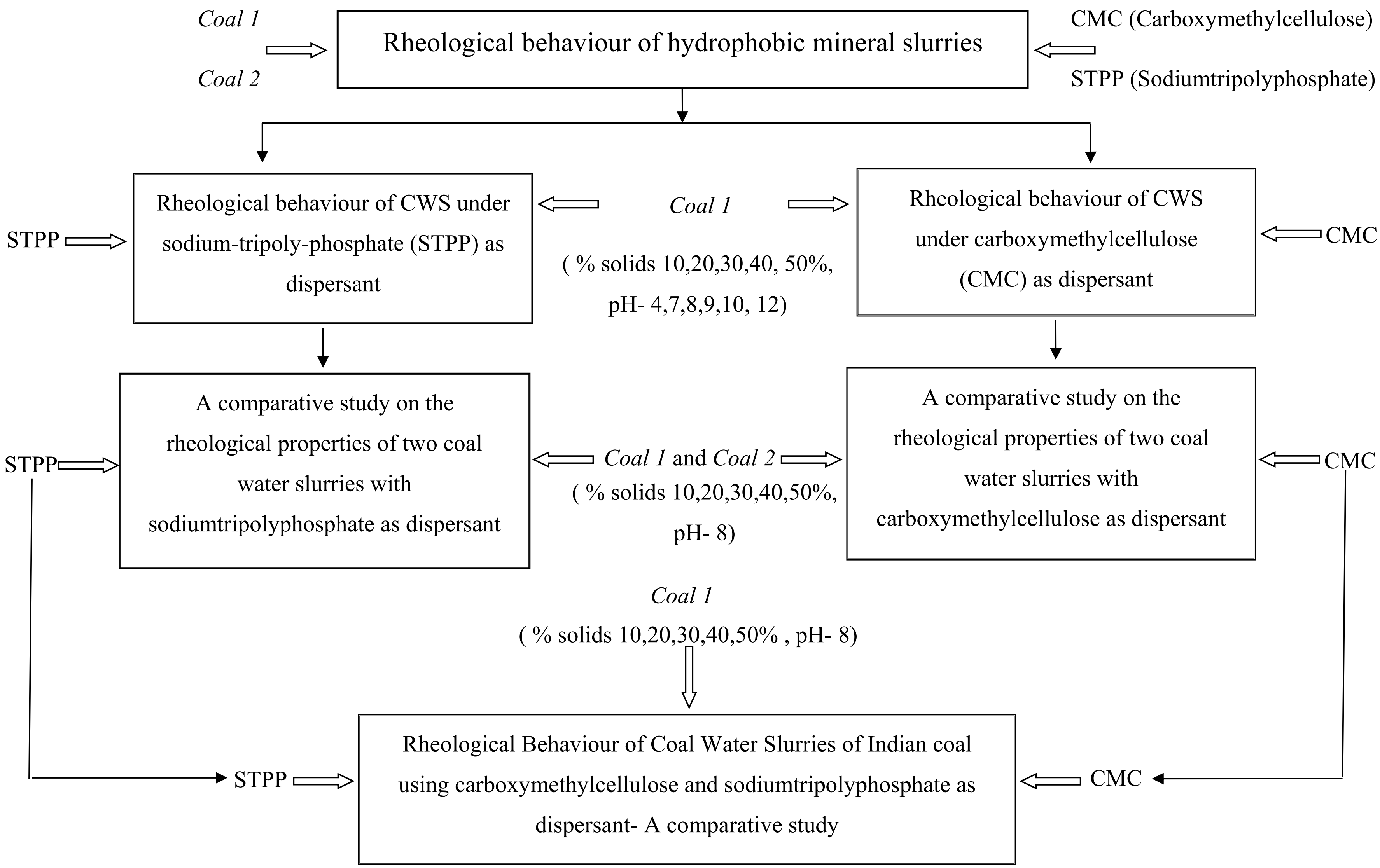


Figure 6. Schematic diagram of research plan

CHAPTER-4

CHAPTER-4

RESULTS AND DISCUSSION

4.0 Introduction

Physical and chemical properties of the coal constituents play crucial role on rheological behaviour of the coal water slurries. Understanding the nature of chemical constituents and their surface characteristics of coal is an important step in selection of proper chemical additive in manipulation of rheological properties of CWS.

This chapter describes the characterization of coal and characterization of dispersant on the coal using various characterization tools namely proximate and ultimate analysis to determine the nature of coal, X-ray diffraction for phase identification, scanning electron microscopy (SEM) with EDS analysis for morphology, chemical nature of the particles, the SEM- elemental mapping for distribution of chemical species in the coal sample. The suitability and effectiveness of the dispersant for the type of coal is discussed by the zetapotential and the turbidity measurements.

4.0.1 Characterization of coal sample

For the coal, the proximate and ultimate analysis shows that the ash constituents, moisture, volatile matter and HGI are comparatively more in *Coal 2* than the C*oal 1*. Consequently, gross calorific value is more for *Coal 1*. Hence it is confirmed that *Coal 1* is relatively better rank than C*oal 2*. (Table 2).

For *Coal 1,* the XRD analysis revealed the presence of minerals such as quartz, kaolinite, pyrite and montmorillonite as the dominant phases in the ash content as shown in Fig.7. The morphology and the chemical distribution of phases are identified using Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Spectroscopy (EDS). An SEM image of coal sample is shown in Fig.8. The presence of flaky and equiaxed particles which are considered to be rich in carbonaceous and ash bearing ones respectively can be seen in the figure. Also, SEM-EDS analysis presented in Fig.9 confirms the presence of carbon, Iron, Aluminium, silicon, sulphur and oxygen etc. The analysis also confirms the presence of particles containing both carbonaceous and non-carbonaceous mineral matter. The chemical distribution of phases is found using SEM-EDS elemental mapping. Fig.10 shows the SEM micrograph of coal sample with elemental mapping for oxygen, silicon, aluminium, calcium, iron, sulphur and carbon conforms the presence of both carbonaceous and non-carbonaceous mineral matter (quartz, kaolinite, pyrite and montmorillonite etc.) in the coal.

Table 2: Proximate and ultimate analysis of the coal samples (*Coal 1* and *Coal 2*)

ADB-Air Dry Basis	***Coal 1***	***Coal 2***
Proximate Analysis (wt.% as received)		
Moisture (ADB)	0.8	2.5
Ash (ADB)	30.2	36.4
Volatile Matter (ADB)	19.9	23.5
Fixed Carbon (ADB)	49.1	37.6
Gross Calorific Value (Kcal/Kg)	5537	4873
Hardgrove Grindability Index	68	70
Ultimate Analysis (wt.% as received)		
Carbon	59.7	49.8
Hydrogen	2.8	3.6
Nitrogen	1.1	0.8
Oxygen	2.9	7.8
Sulphur	0.3	0.6

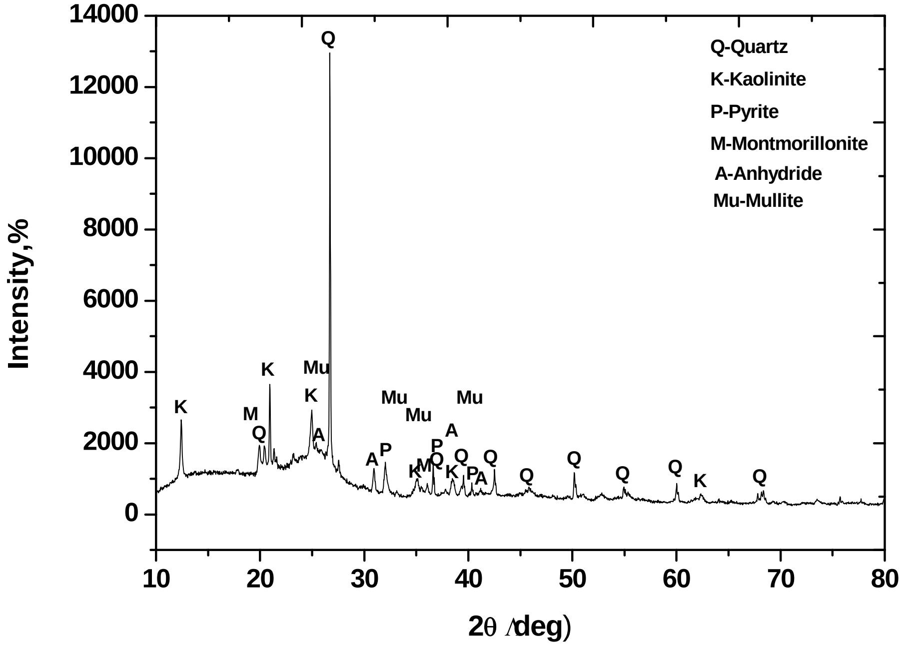


Figure 7. XRD pattern of the *Coal 1* sample.

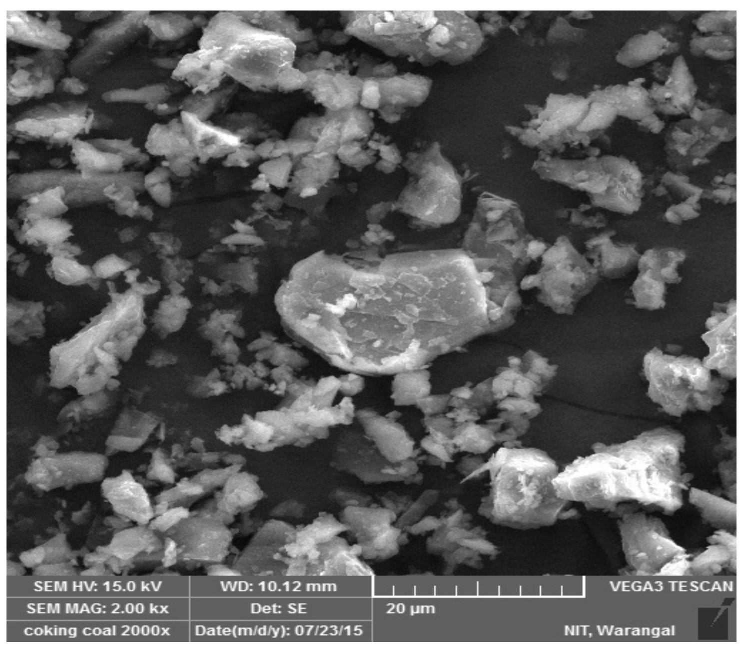
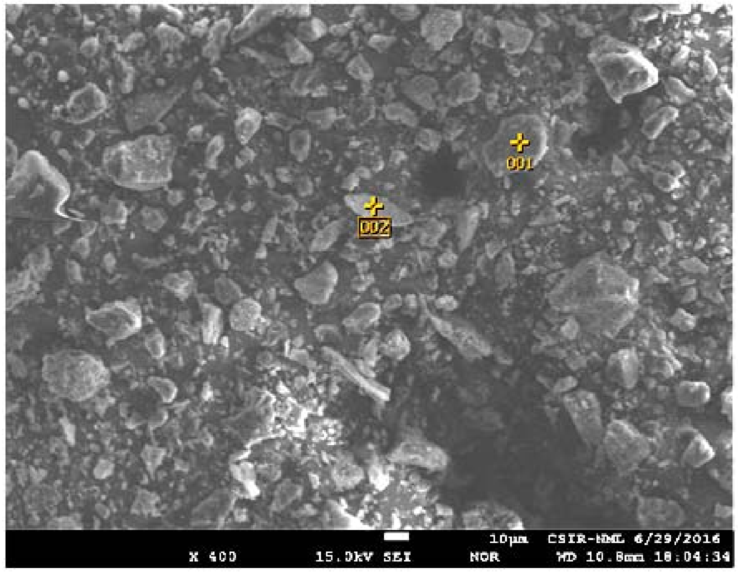


Figure 8. SEM-micrograph of *Coal 1* sample.



Location 001:
Fe-0.98, O-16.68, C-79.66, Al-0.84, Si-1.08, S-0.33 & Cl-0.44
Location 002:
Fe-28.99, O-6.21, C-53.58, Al-3.30, Si-6.40, S-0.30, Cl-0.48 & Mg-0.75

Figure 9. Scanning electron micrograph of *Coal 1* sample and the EDS analysis results at two locations (elements given in weight percentage).

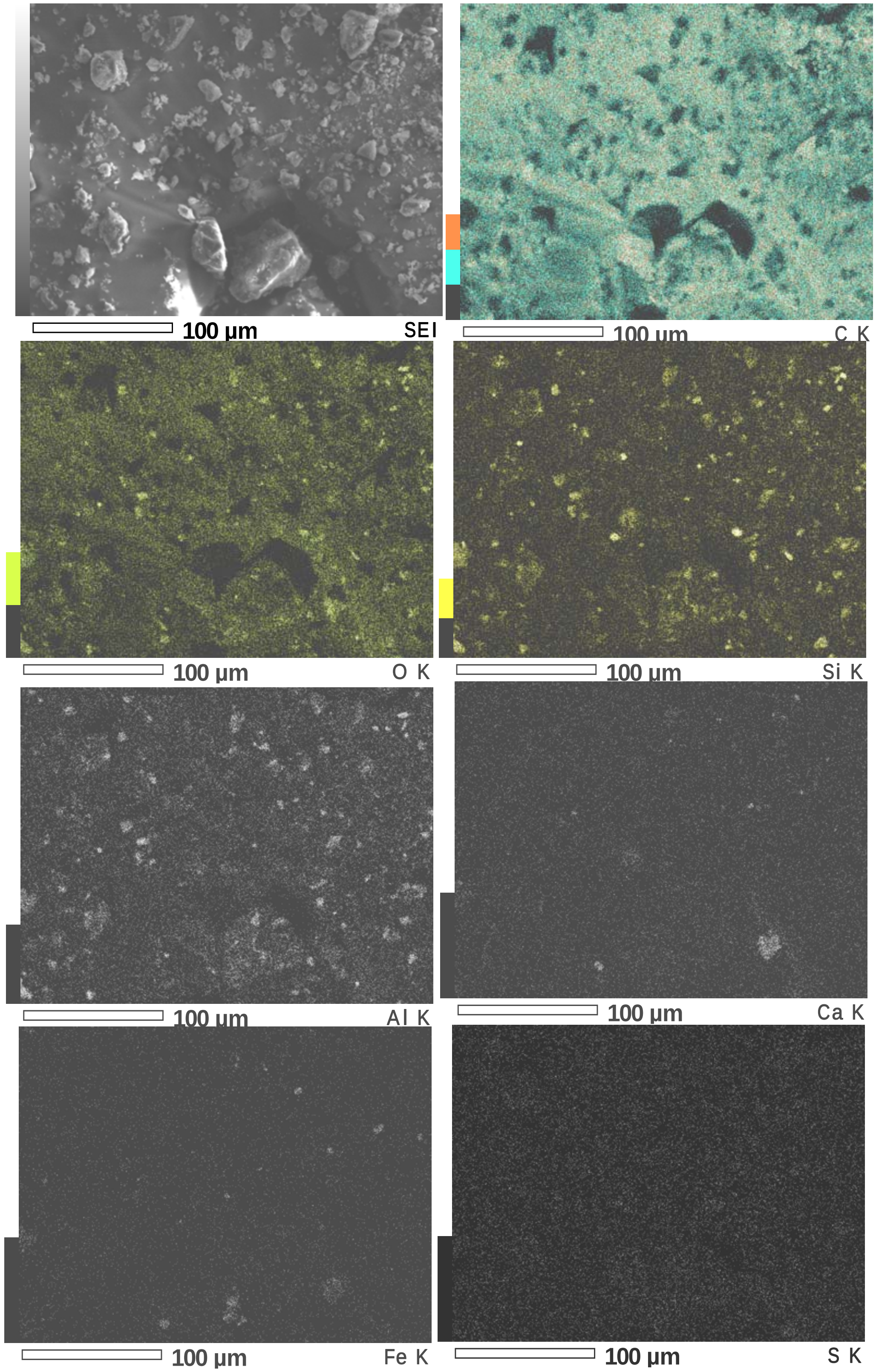


Figure 10. Scanning electron micrograph of *Coal 1* sample & its elemental mapping of carbon, oxygen, silicon, aluminium, calcium, iron and sulphur.

4.0.2 Zeta potential and Turbidity Evaluation

The knowledge on zetapotential and turbidity of the coal in the presence of chemical additive or dispersant can greatly help us in the manipulation of rheological properties of the CWS. Zetapotential and turbidity measurements were carried out on the coal samples (*Coal 1* and *Coal 2*) in the presence of carboxymethylcellulose (CMC) and sodiumtripolyphosphate (STPP) addition. In general, the rheological behaviour of a CWS is greatly influenced by the zetapotential of the coal surface. A higher negative value of zeta potential can lead to minimum viscosity and good dispersion which are beneficial for the slurry transportation [51].

4.0.3 Zetapotential and Turbidity of the coal samples under CMC as dispersant

Zetapotential and turbidity measurements were carried out on the *Coal 1* and *Coal 2* samples with and without the addition of CMC as dispersant. The results are presented in Fig.11 & Fig.12 respectively. A continuous decrease of zetapotential with addition of dispersant was observed for both samples. A lower zetapotential and a sharp decrease in zetapotential was observed for *Coal 1* in comparison to *Coal 2.* A gradual increase in the turbidity was observed for both the coals. Higher and gradual increase in the turbidity was observed for *Coal 1* in comparison to *Coal 2*. Overall, a decrease of zetapotential and an increase of turbidity of the two coal samples with an increase in dispersant dosage indicate the suitability of CMC as a dispersant in preparation of CWS using the coal samples. Moreover, marginal improvement of dispersant effect was seen for the *Coal 1* in comparison to that of *Coal 2*.

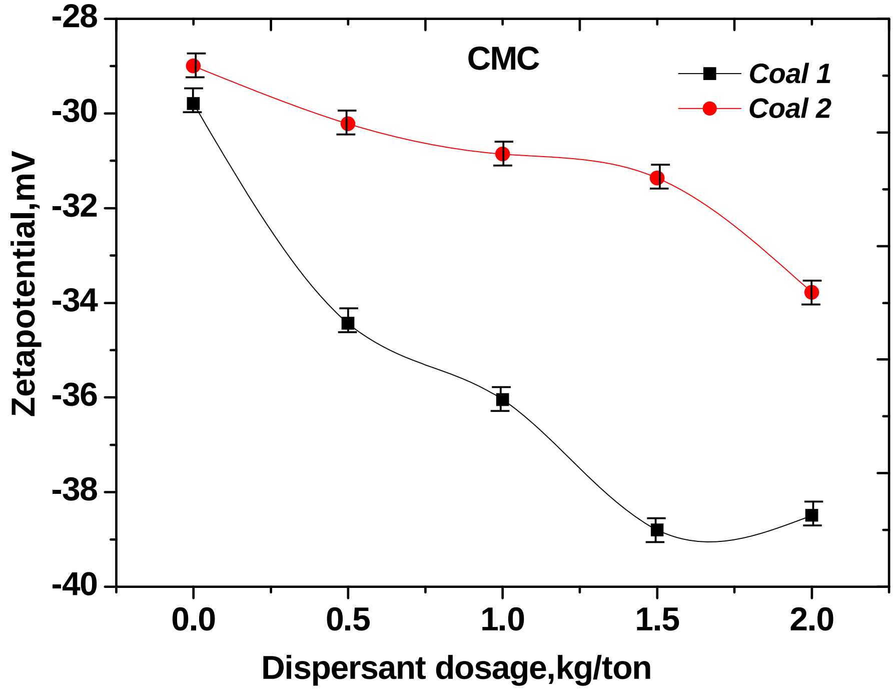


Figure 11. Zetapotential of the *Coal 1* and *Coal 2* as a function of CMC as a dispersant.

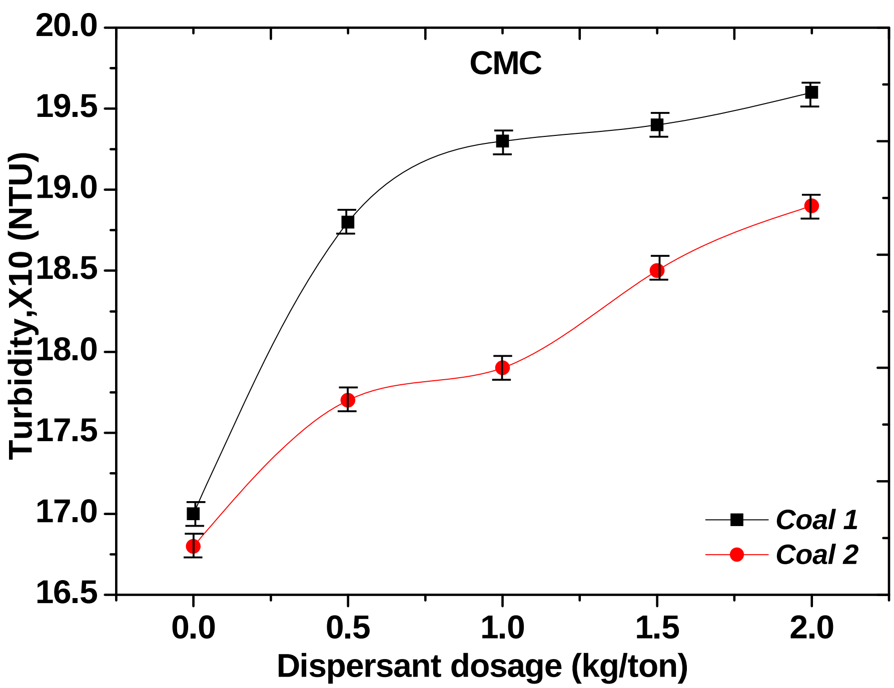


Figure 12. Turbidity of the *Coal 1* and *Coal 2* as a function of CMC as a dispersant.

4.0.4 Zetapotential and Turbidity of the coal samples under STPP as dispersant

The rheological behaviour of the CWS was greatly influenced by the zetapotential and turbidity of the coal. Zetapotential and turbidity measurements were carried out on the *Coal 1* and *Coal 2* samples with and without the addition of STPP as dispersant. The results were presented in Fig.13 & Fig.14 respectively. A gradual increase in the turbidity and a continuous decrease in zetapotential with addition of dispersant was observed for both the coal samples. Higher and gradual increase in the turbidity was observed for *Coal 1* in comparison to *Coal 2*. A lower zetapotential and a sharp decrease in zetapotential was observed for the *Coal 1* in comparison to *Coal 2*. Overall, a decrease of zetapotential and an increase in turbidity of the two coal samples with an increase in dispersant dosage indicate the suitability of STPP as a dispersant in preparation of CWS using the coal samples.

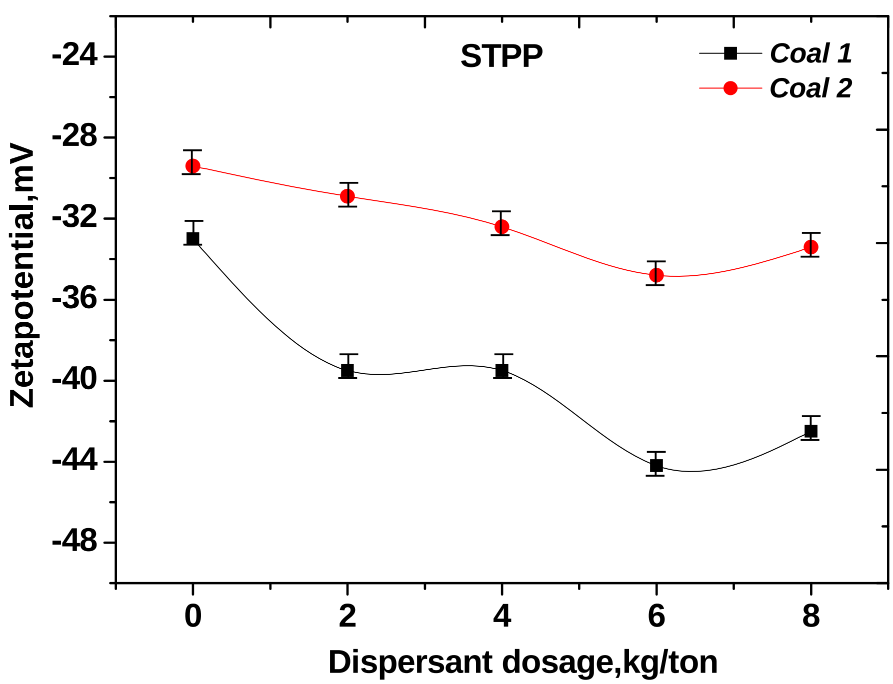


Figure 13. Zetapotential of the *Coal 1* and *Coal 2* as a function of STPP as a dispersant.

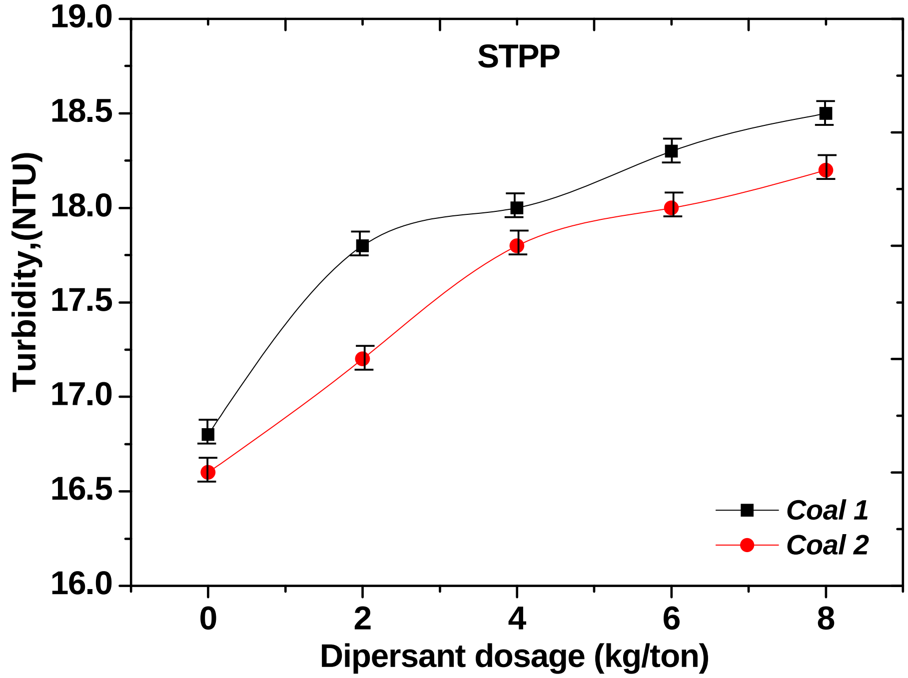


Figure 14. Turbidity of the *Coal 1* and *Coal 2* as a function of STPP as a dispersant.

4.0.5 Comparison of zetapotential and turbidity of coal sample for CMC and STPP as dispersant

Zetapotential and turbidity measurements were carried out on the coal sample (*Coal 1*) in the presence of dispersants (CMC and STPP) addition. The results were presented in Fig.15 & Fig.16 respectively. A continuous increase in the turbidity and a gradual decrease in zetapotential with addition of dispersant was observed for both the dispersants. A higher turbidity and a lower zetapotential were observed for CMC in comparison to STPP, which indicate that the CMC is much effective in dispersing CWS in comparison to STPP as a dispersant. Overall, a decrease in zetapotential and an increase in turbidity in the presence of dispersant dosage indicate the suitability of CMC and STPP as a dispersant in preparation of CWS using the coal sample.

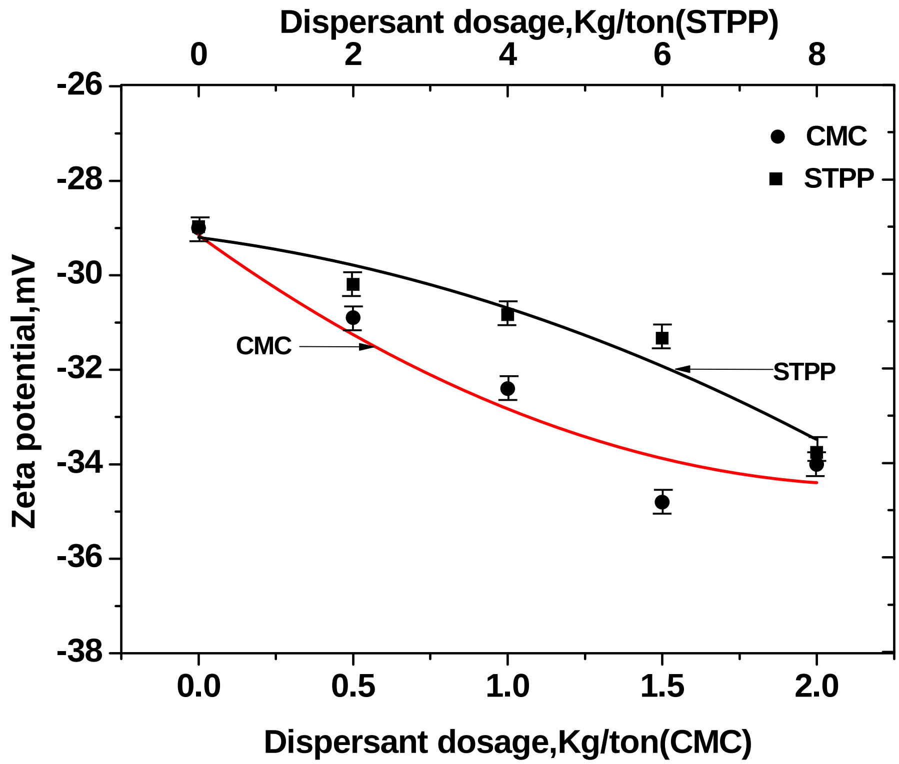


Figure 15. Zetapotential of the *Coal 1* as a function of CMC and STPP as a dispersant

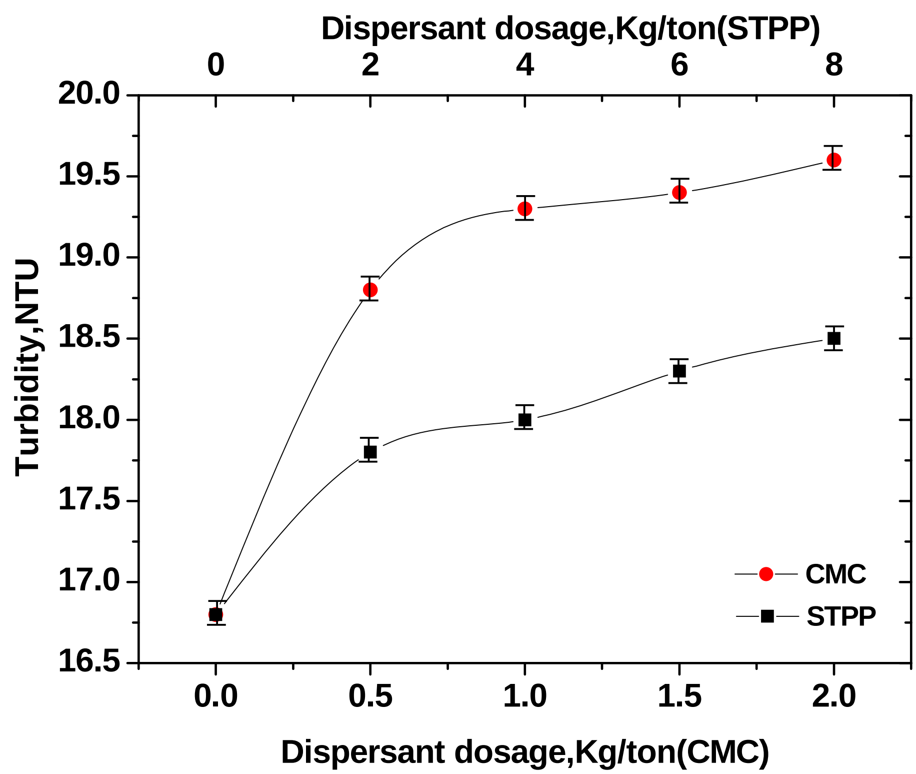


Figure 16. Turbidity of the *Coal 1* as a function of CMC and STPP as a dispersant.

4.0.6 Summary

Proximate and ultimate analysis confirms lower ash content in *Coal 1* in comparison to *Coal 2*. The XRD analysis revealed the presence of minerals such as quartz, kaolinite, pyrite and montmorillonite as dominant phases as ash bearing elements in the coal. SEM morphology and its chemical analysis by Energy Dispersing Spectroscopy (EDS) conforms the presence of the particles containing both carbonaceous and non-carbonaceous mineral matter. The zetapotential and turbidity measurements indicated the suitability of CMC and STPP as dispersant for the CWS prepared using the two coal varieties. The dispersant addition is much effective for *Coal 1* in comparison to *Coal 2*. Zetapotential and turbidity confirms that the CMC is much effective dispersant in comparison to STPP for the coal variety tested.

4.1 Rheological behaviour of CWS under Sodiumtripolyphosphate (STPP) as dispersant

4.1.1 Introduction

A good understanding of the rheological properties of coal water slurries is essential for design and optimisation of the processes. The flow characteristics of the coal water suspensions depends on (1) physical and chemical properties of the coal such as ash content, the amount of inherent water, the degree of coal oxidation, and the quantity of surface active functional groups;(2) the volume fraction, ϕ, of the suspension; (3) the particle size range and its distribution, (4) interparticle interactions in the suspension and their effects of pH and the chemical additives etc. Chemical additives are important ingredients in reducing the viscosity, maintaining fluidity and improving the stability of CWS by introducing the electrostatic or steric repulsions or increasing the steric wettability of coal.

This chapter describes the rheological behaviour of CWS in the presence of an anionic dispersant namely Sodiumtripolyphosphate (STPP, Chemical Formula: $Na_5P_3O_{10}$) using Indian low rank coal variety (*Coal 1*) mined in Jamadoda, Jharkhand state. For all CWS, the effect of different solids loading (10%, 20%, 30%, 40% and 50%), dispersant dosages (2.0, 4.0, 6.0 and 8.0 kg/ton) and slurry pH (4,7, 8, 9, 10 and 12) on flow behaviour is studied at shear rate in the range of 60-160 s^{-1}. The rheology data was fitted for power law model (refer equation (1) as shown in Chapter 2, Section 2.1.2.1) and the flow behaviour index was estimated to identify the rheological nature as a function of solids loading, dispersant dosage and pH of the of the slurry.

4.1.2 Effect of solids concentration of CWS for STPP as dispersant

The rheological data plotted as the variation shear stress with respect to dispersant dosage (constant shear rate curves) are presented in Fig.17 (a-f) - Fig.21 (a-f). Upon closer examination of the plots, the rheological behaviour of all suspensions at different pH values is found to be Non-Newtonian. For the lower percentage of solids (10%, 20%), the shear stress versus shear rate relations are almost similar in nature and magnitude with respect to dispersant dosage for all pH values (Fig.17 (a-f) - Fig.18 (a-f)). On the other hand, the dispersant effect is much pronounced for the higher solid loadings (30%, 40% and 50%). For an increase in solids concentration, an increase in the shear stress values is seen (Fig.19 (a-f)-

Fig.21 (a-f)). Due to the shear interaction of particles and significant friction among them can be seen as the reason for the same [11,53].

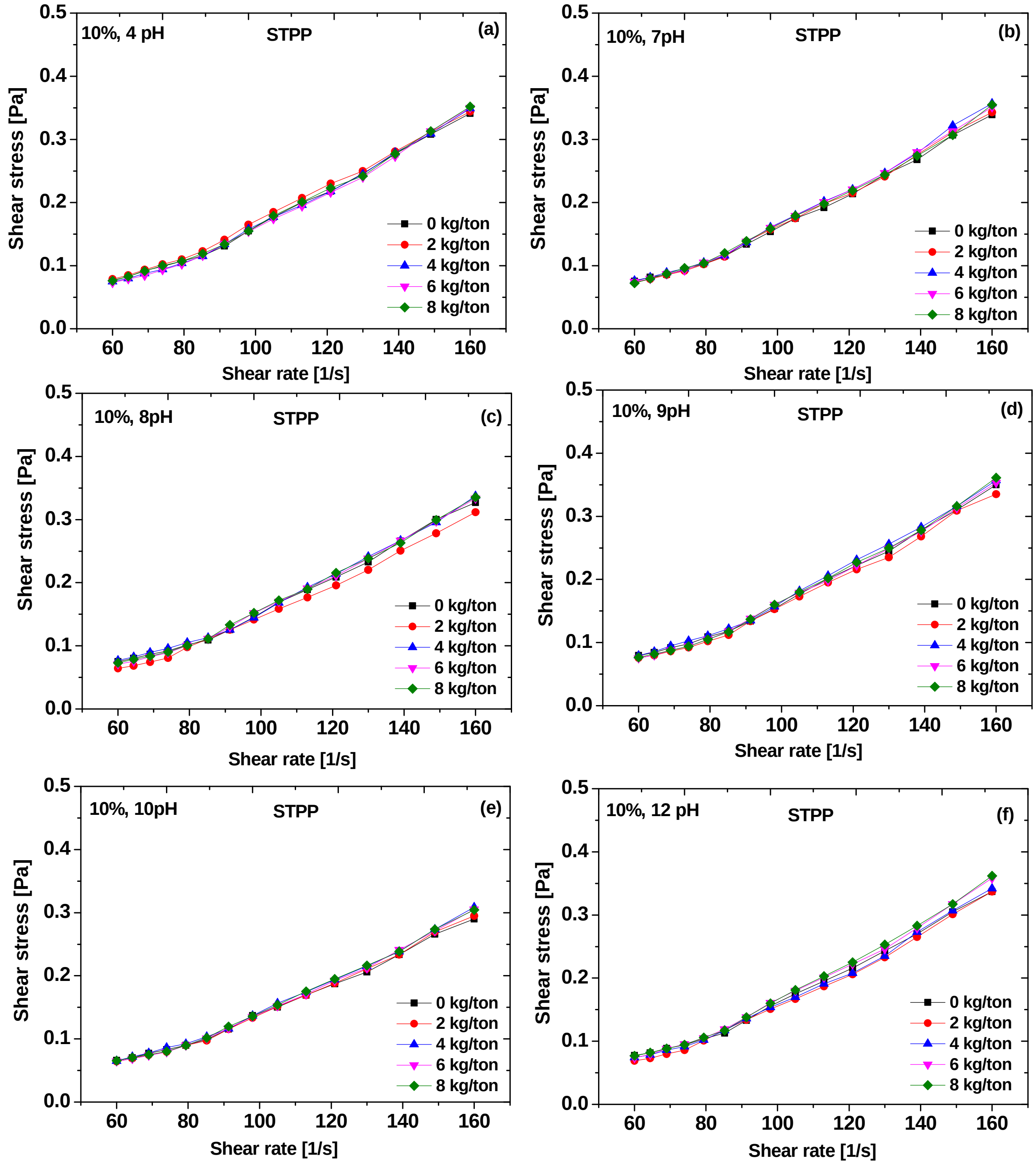


Figure 17. Shear stress versus shear rate on CWS of 10% solids concentration at pH (a) 4, (b)7, (c) 8, (d) 9, (e) 10 and (f) 12 for STPP as dispersant

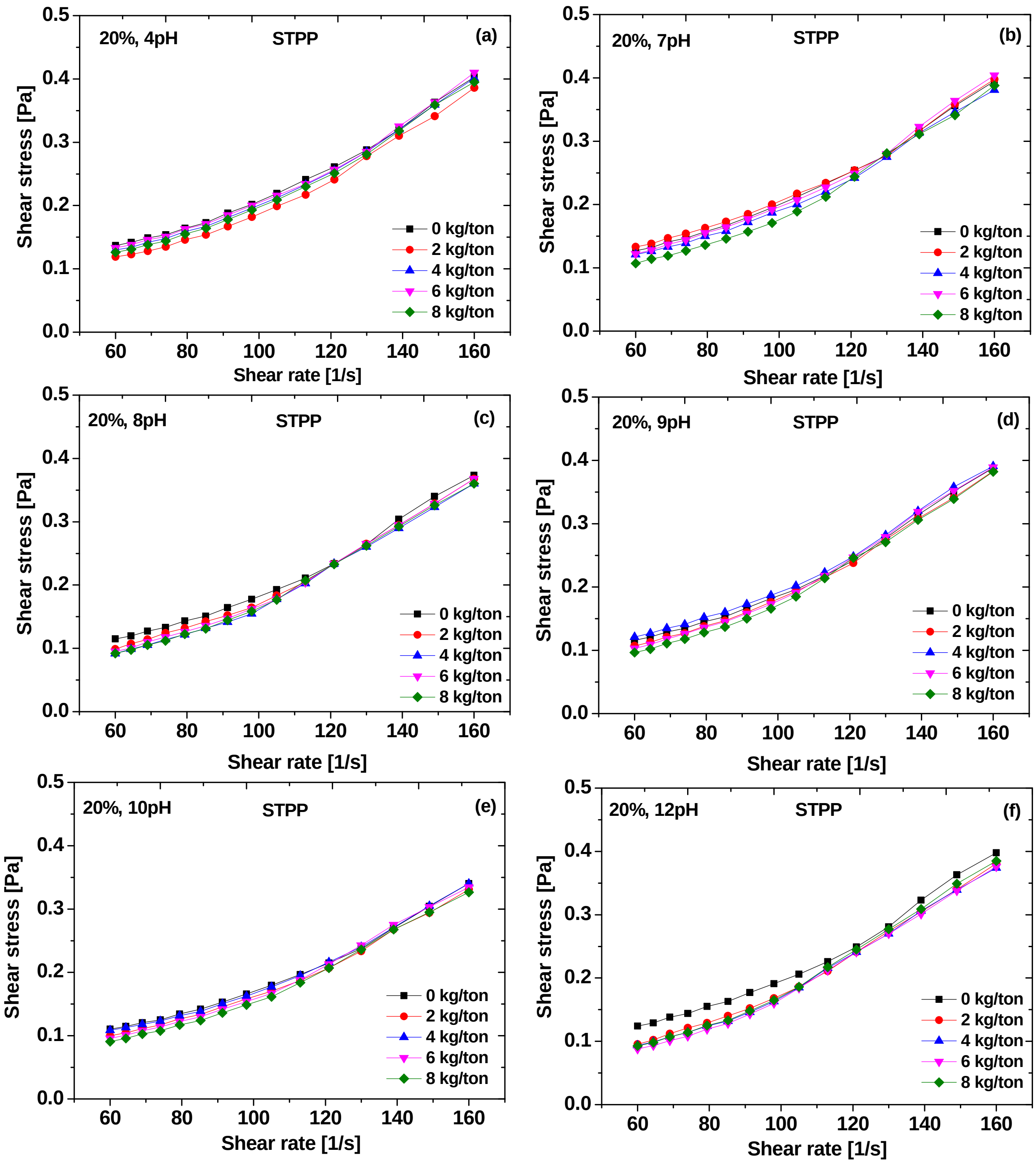


Figure 18. Shear stress versus shear rate on CWS of 20% solids concentration at pH (a) 4, (b)7, (c) 8, (d) 9, (e) 10 and (f) 12 for STPP as dispersant

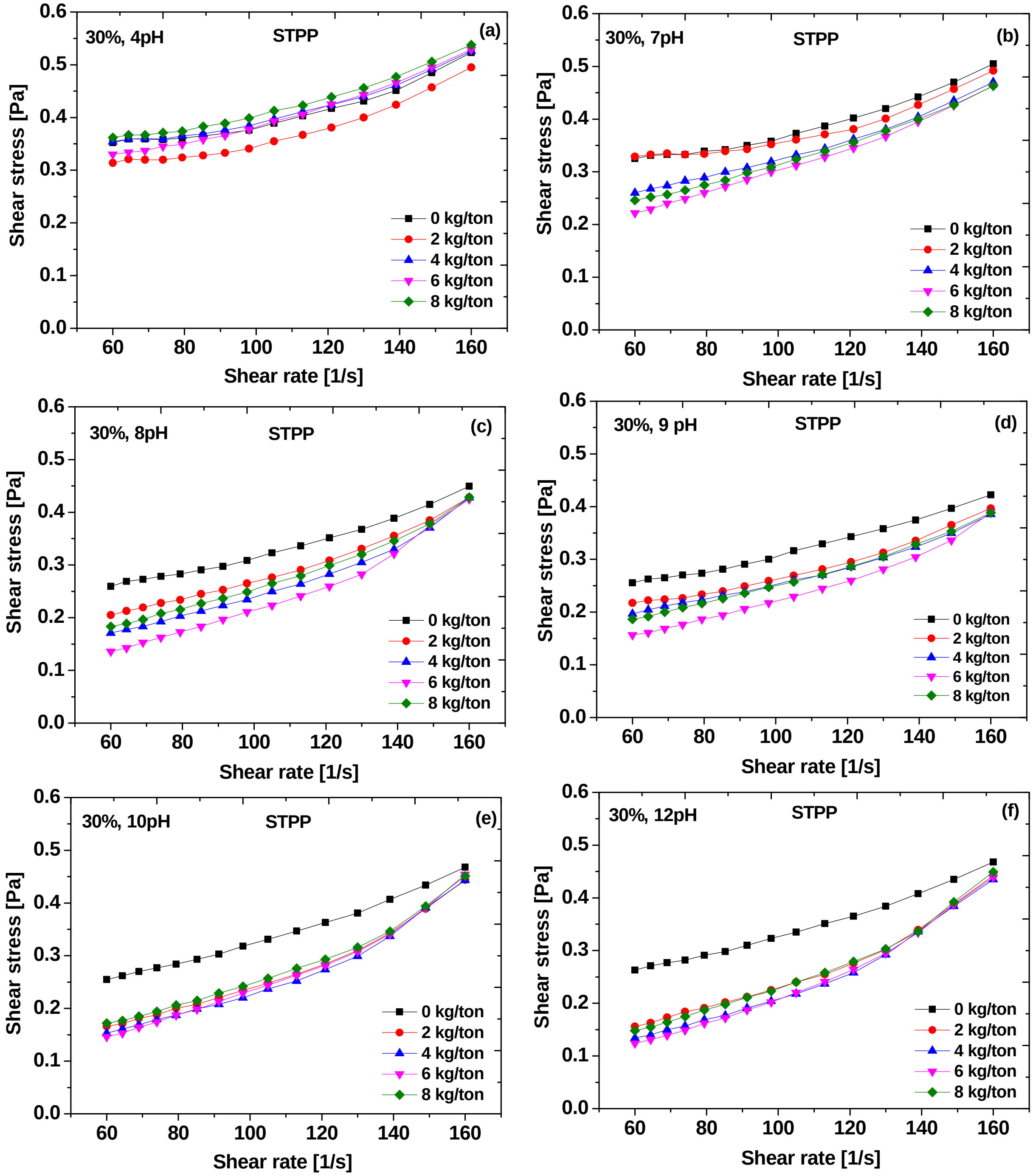


Figure 19. Shear stress versus shear rate on CWS of 30% solids concentration at pH (a) 4, (b)7, (c) 8, (d) 9, (e) 10 and (f) 12 for STPP as dispersant

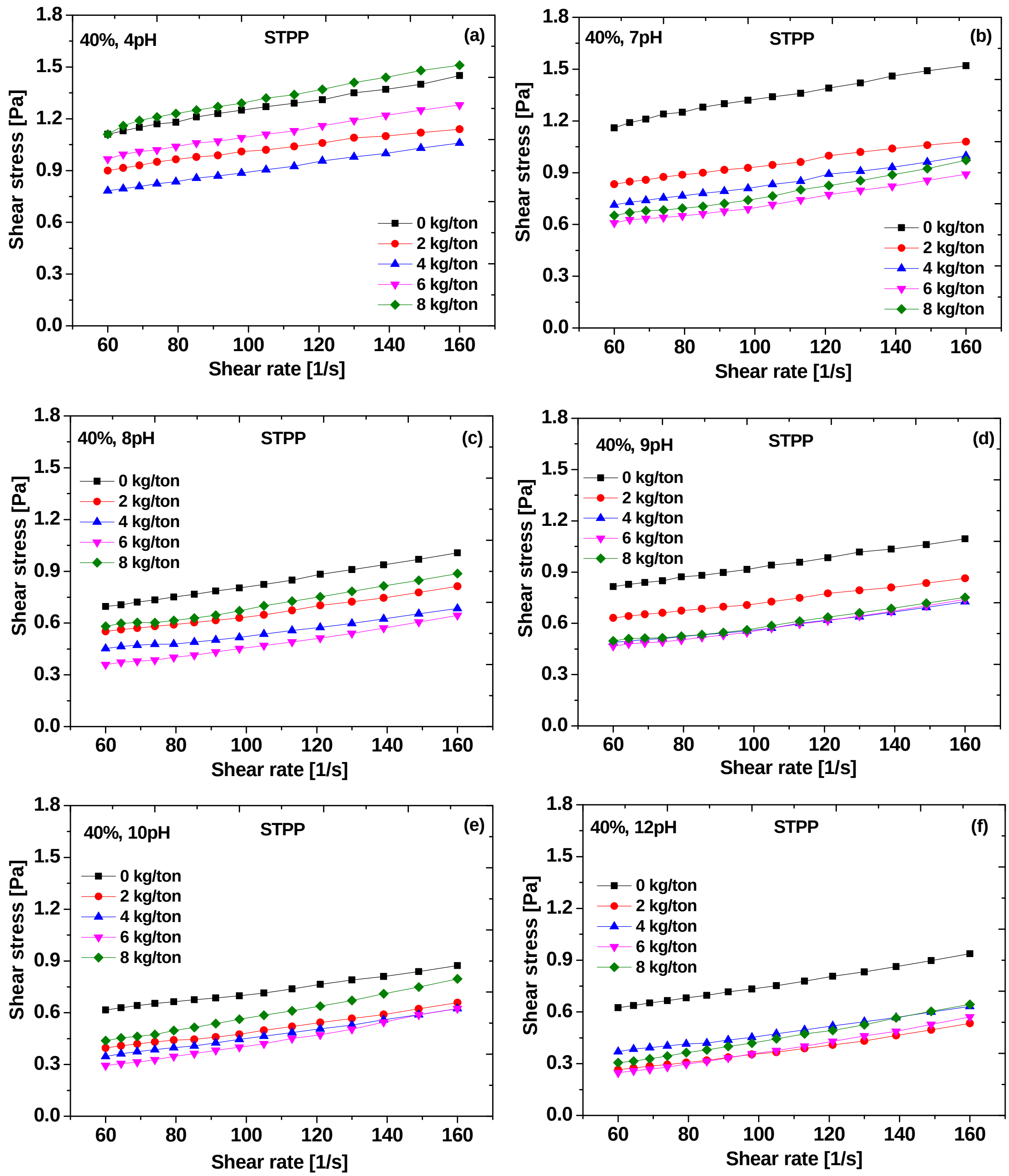


Figure 20. Shear stress versus shear rate on CWS of 40% solids concentration at pH (a) 4, (b)7, (c) 8, (d) 9, (e) 10 and (f) 12 for STPP as dispersant

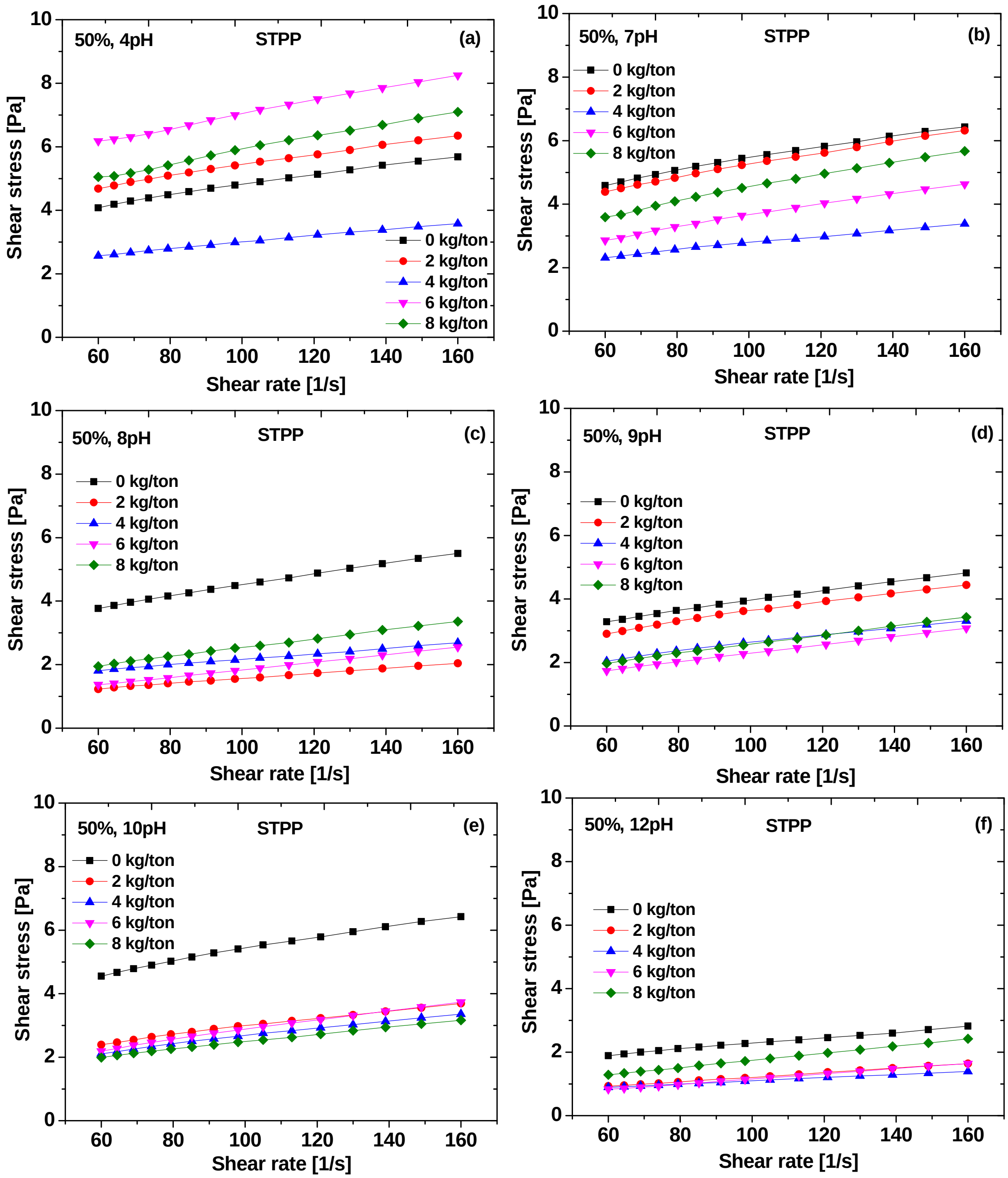


Figure 21. Shear stress versus shear rate on CWS of 50% solids concentration at pH (a) 4, (b)7, (c) 8, (d) 9, (e) 10 and (f) 12 for STPP as dispersant

4.1.3 Effect of dispersant on CWS for STPP as dispersant

To understand the effect of dispersant dosage on shear rate, the rheological data plotted as the variation of shear stress with respect to dispersant dosage (constant shear rate curves) are presented in Fig.22 (a-f) – Fig.26 (a-f). The dispersant dosage has no significant effect on the shear stress values for 10% and 20% solid loading for all pH values as observed in Fig.22 (a-f) – Fig.23 (a-b). For 30% solid loading (Fig.24 (a-f)), at pH of 4, the shear stress attains minima at dispersant dosage of 2 kg /ton for all shear rates tested. Minimal shear stress values are seen for 6 kg / ton dispersant loading for pH values 7,8 and 9 except the case with pH =9 & shear rate 160 s^{-1}. For the highest shear rate tested (160 s^{-1}), the dispersant has no effect as the magnitude of shear stress change is minimal for this solid loading. For pH = 10 & 12, the variations of shear stress with respect to dispersant dosage for the different shear rates are similar. For these two cases, minimum shear stress value is seen at 6 kg/ ton dispersant loading for the shear rates of 60 and 74.1 s^{-1}. For the shear rates of 121 & 160 s^{-1}, the minimum shear stress values are seen at 4 kg/ton dispersant loading.

In the case of 40% solid loading (Fig.25 (a-f)), for pH values 4 and 7, the dispersant loadings at which shear stress values are minimum are 4 kg/ton and 6 kg/ton respectively. For pH of 9, for the lower shear rate values (60, 74 and 91.3), shear stress values are minimum at 6 kg/ton dispersant loading. For shear rate 160 s^{-1} and 4 kg/ton dispersant loading, the shear stress is minimum. For other pH values, minimum shear stress values are seen at a dispersant dosage of 6 kg per ton. Two local minima values are seen in the case of the highest pH value, i.e., 12. Minimum shear stress values are seen for the lower shear rate values (60, 74 and 91.3) at dispersant dosage 6 kg/ ton while the same are seen with 2 kg/ton for the higher shear rate values (120 and 160 s^{-1}).

For 50% solid loading (Fig.26 (a-f)), the minimum shear stress values obtained for pH values of 4, 7, 9 and 10 are 4, 4, 6 and 8 kg per ton of dispersant respectively. It indicates the combined effect of pH and dispersant dosage on the rheological behaviour of the 50% CWS. At pH 8 and 12, two minima values for each case are seen at 2 kg/ton and 6 kg/ton for all shear rates. For pH of 12, minimum shear stress values are seen at 6 and 4 kg per ton of dispersant for the lower (60, 74, 91.3 s^{-1}) and higher (121, 160 s^{-1}) shear rates respectively.

For a given percent solids and pH, an increase of shear stress was observed from 6 kg per ton dispersant loading to 8 kg per ton. This can be due to agglomeration of particles in the slurry after attaining saturation limit of the dispersant. The excessive dispersant dosage can increase the ionic strength of the slurry which results in formation of strong electrical double layers around the solid particles and thereby obvious reduction in the electrostatic repulsive forces among the particles. As a result, the shear stress values increase with increase of dispersant loading after the saturation limit [52,54]. For a given percent solids and dispersant dosage, a decrease in shear stress is seen at higher shear rate values. This is due to the continuous breakdown of structure in the slurry or continuous and sudden breakdown of aggregates in the slurry [53].

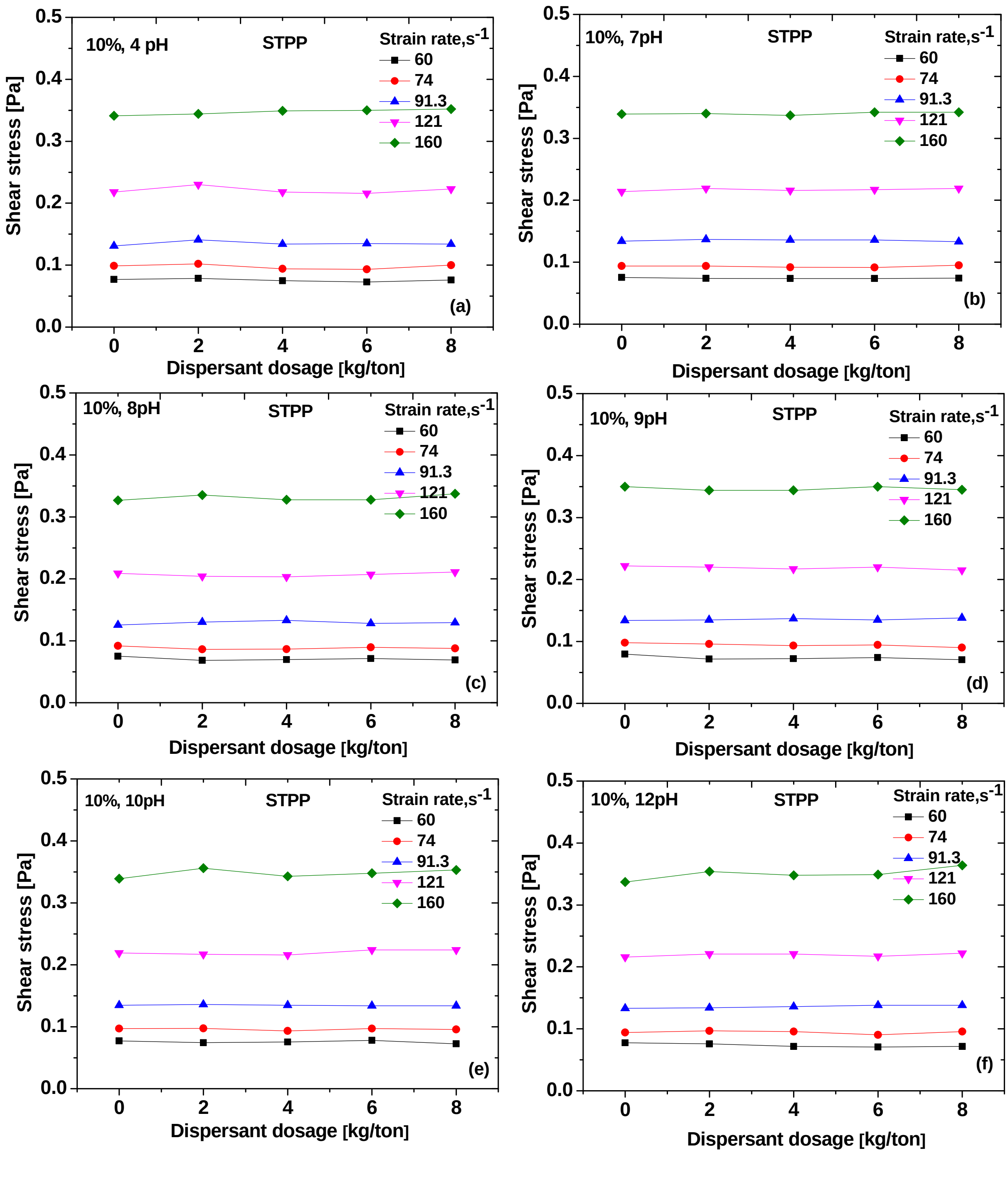


Figure 22. Effect of STPP dispersant dosage on 10% solid loadings at pH of (a) 4, (b) 7, (c) 8, (d) 9, (e) 10 and (f) 12.

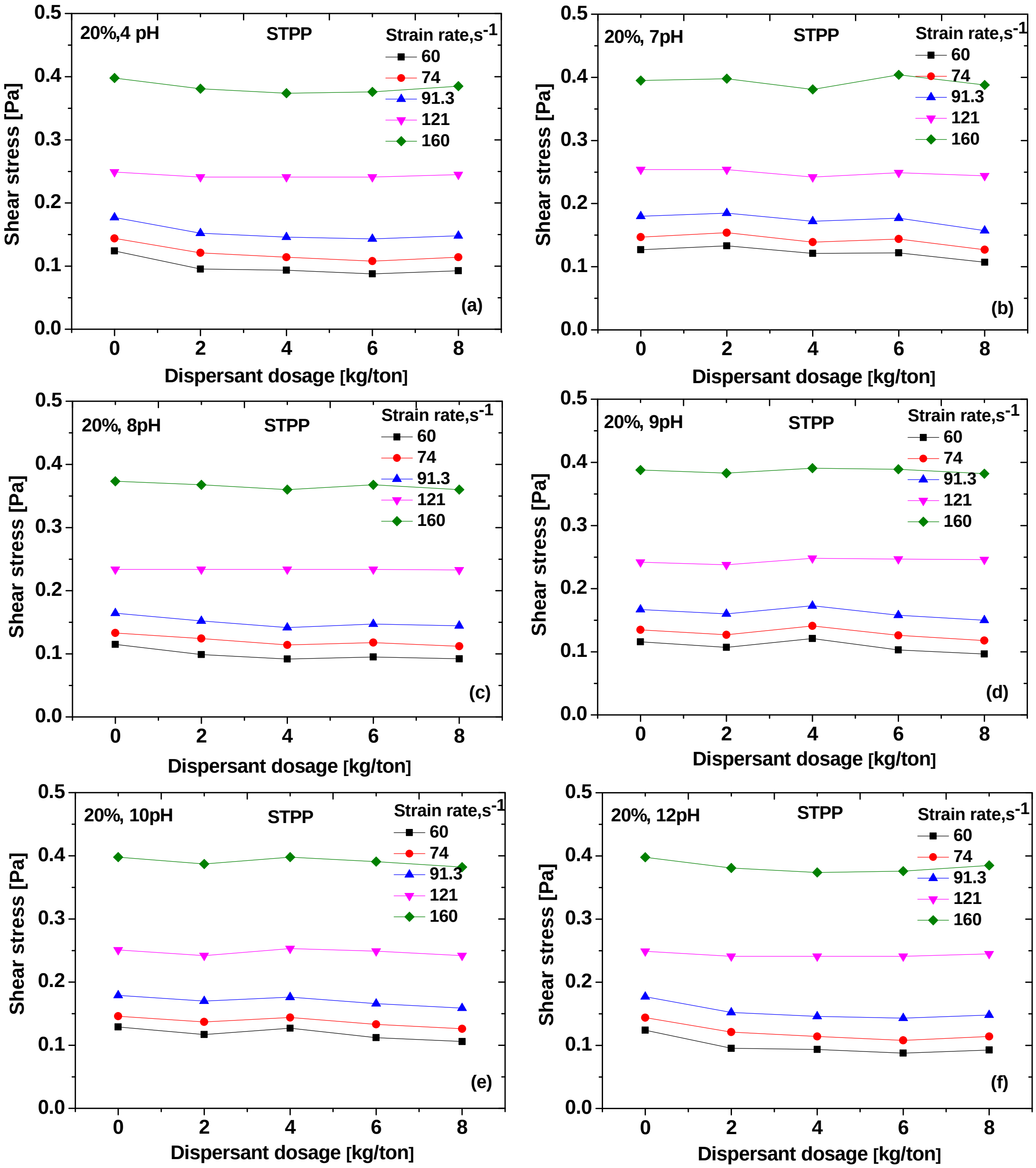


Figure 23. Effect of STPP dispersant dosage on 20% solid loadings at pH of (a) 4, (b) 7, (c) 8, (d) 9, (e) 10 and (f) 12.

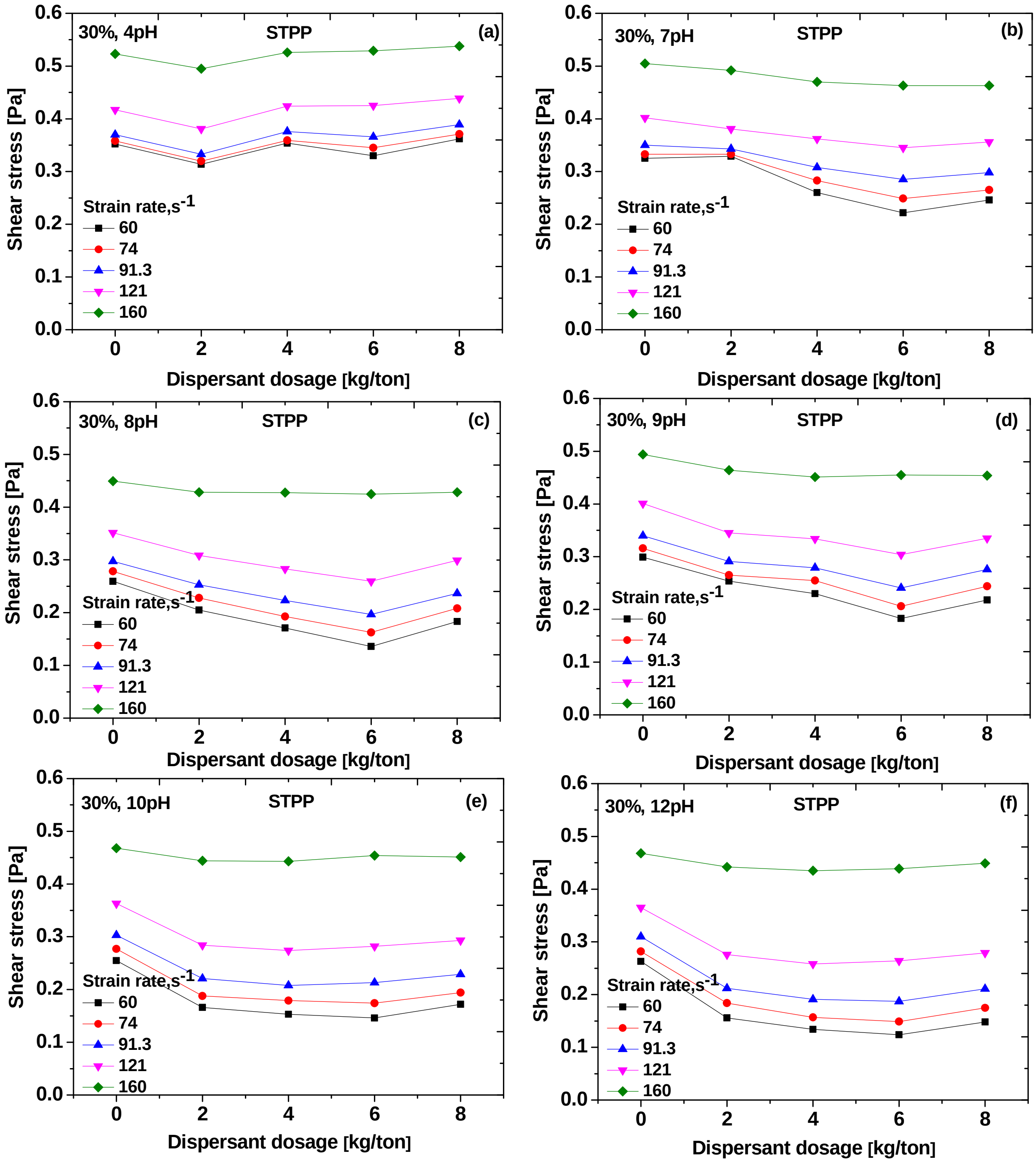


Figure 24. Effect of STPP dispersant dosage on 30% solid loadings at pH of (a) 4, (b) 7, (c) 8, (d) 9, (e) 10 and (f) 12.

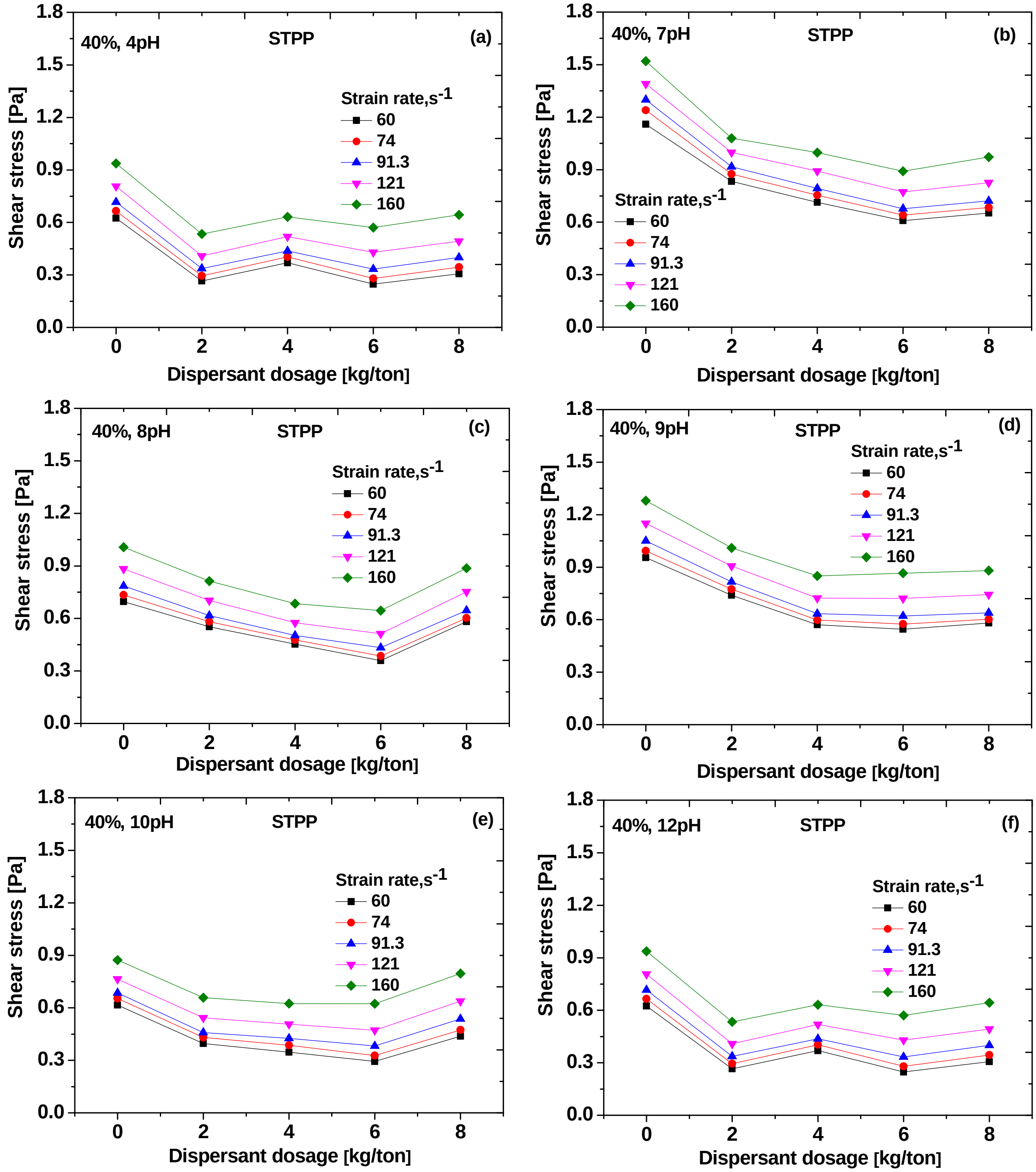


Figure 25. Effect of STPP dispersant dosage on 40% solid loadings at pH of (a) 4, (b) 7, (c) 8, (d) 9, (e) 10 and (f) 12.

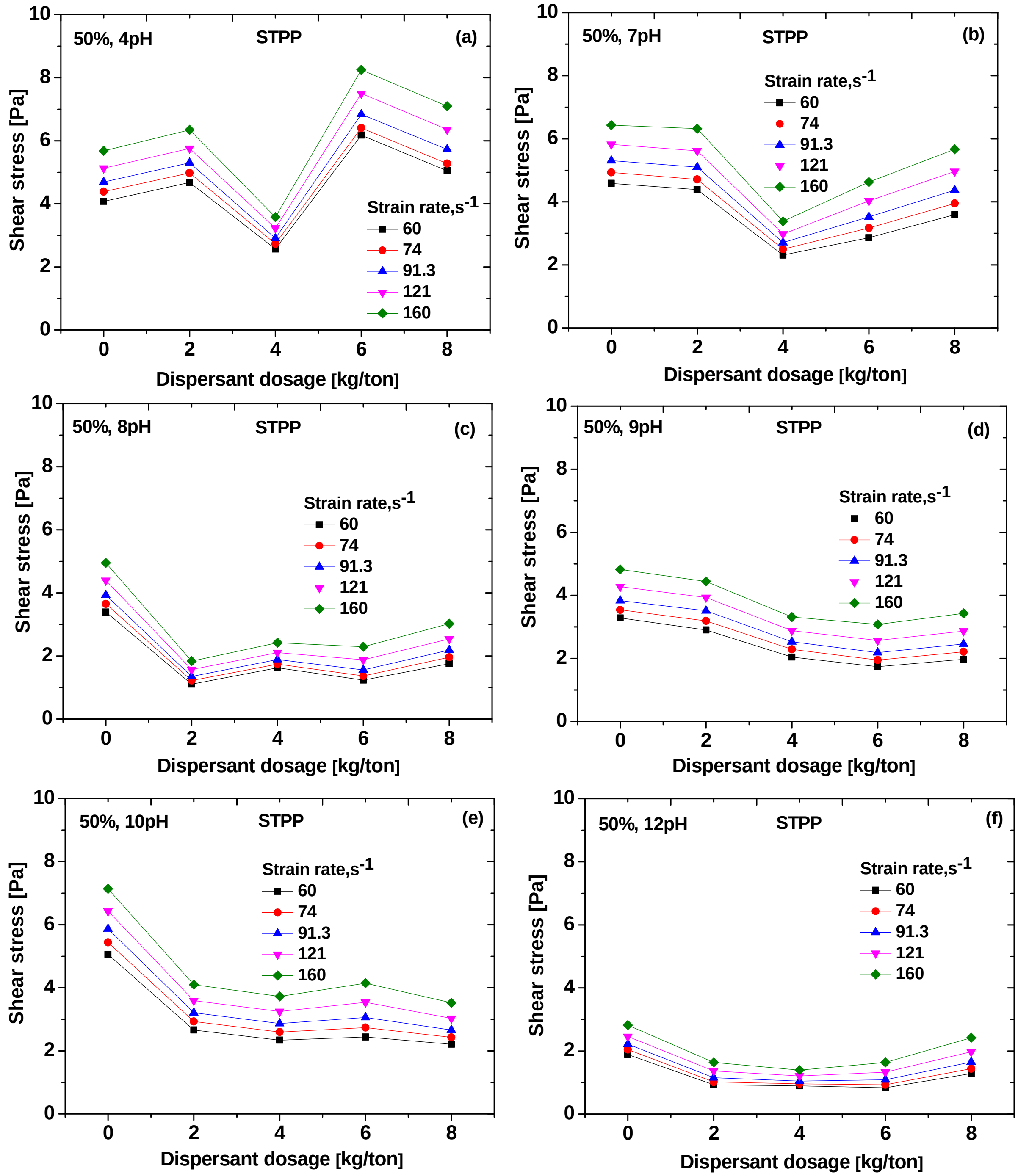


Figure 26. Effect of STPP dispersant dosage on 50% solid loadings at pH of (a) 4, (b) 7, (c) 8, (d) 9, (e) 10 and (f) 12.

4.1.4 Effect of pH on CWS for STPP as dispersant

To understand the effect of pH on shear rate values, the rheological data plotted as the variation shear stress with respect to pH (constant shear rate curves) are presented in Fig.27 (a-e) - Fig.31 (a-e). For 10% and 20% solid loadings, the pH has no appreciable effect on the shear stress for a given shear rate as observed in Fig.27 (a-e) – Fig.28 (a-e). For 30% solids loading (Fig.29 (a-e)), the shear stress values almost exhibited decreasing trend in all cases with an increase in pH for all dispersant loadings. In the absence of dispersant, for 40% loading (Fig.30 (a-e)), maximum shear stress values are attained at pH=7 for the five shear rate values. The shear stress values exhibited decreasing trend with respect to pH in the other cases (with the addition of dispersant). The shear stress exhibited two local maximum values for the pH values of 8 and 10 in the case of 50 % solid loading with no dispersant. The local maximum values of shear stress are present for 2 and 4 kg /ton dispersant loading. The decrease of shear stress with respect to pH increase for all individual shear rates is evident for the other dispersant dosages (6 and 8 kg/ton) tested for 50 % solid loading (Fig.31 (a-e)).

The surface chemistry of the suspension particles is crucial in attributing definite rheological properties to the slurry [26]. If the attractive forces among the particles are strong, higher shear forces are required to overcome the friction. Overall, the effect of dispersant at 4 and 6 kg /ton loading is clearly evident in reducing the shear stress values for 30 %, 40% and 50% solid loading for all pH values as the adsorption of dispersant on the surface of the solids contributes to the countering of the attractive forces. The decrease in shear stress for a given shear rate is observed with increase in pH for the 30% and 40% solid loadings is seen. At lower pH values, the presence of H^+ ions in the liquid media can hamper the adsorption of tripolyphosphate ions on the solid media surface. This phenomenon nullifies the effect of the dispersant. On the other hand, at higher pH values, the sufficient adsorption of tripolyphosphate ions on the particulate matter increases the electrostatic repulsions among them and contributes to the decrease in the shear stress values [55,56].

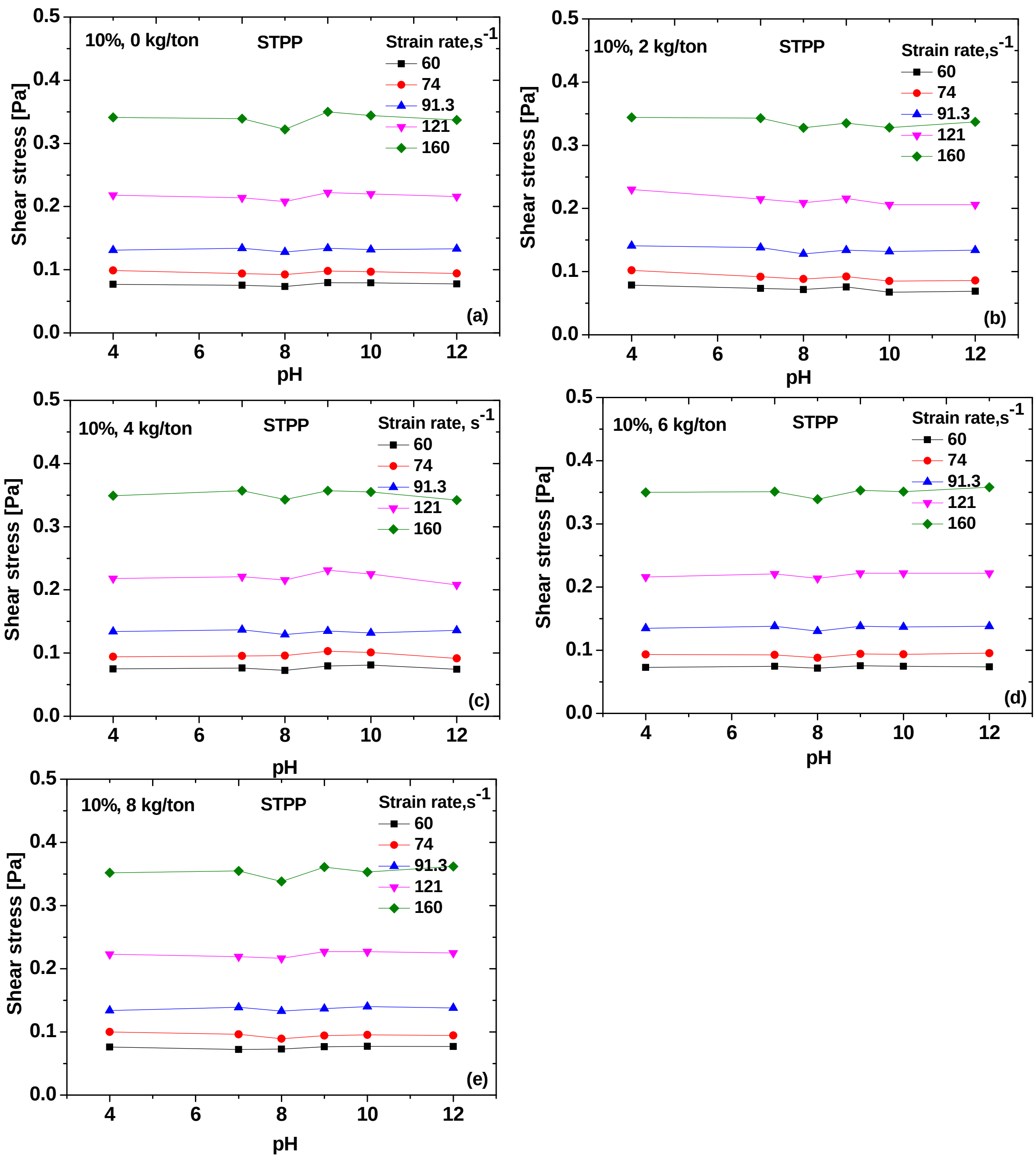


Figure 27. Effect of pH on CWS at 10% solids for different STPP dispersant dosages (a) 0.0 kg/ton, (b) 2.0 kg/ton, (c) 4.0 kg/ton, (d) 6.0 kg/ton and (e) 8.0 kg/ton.

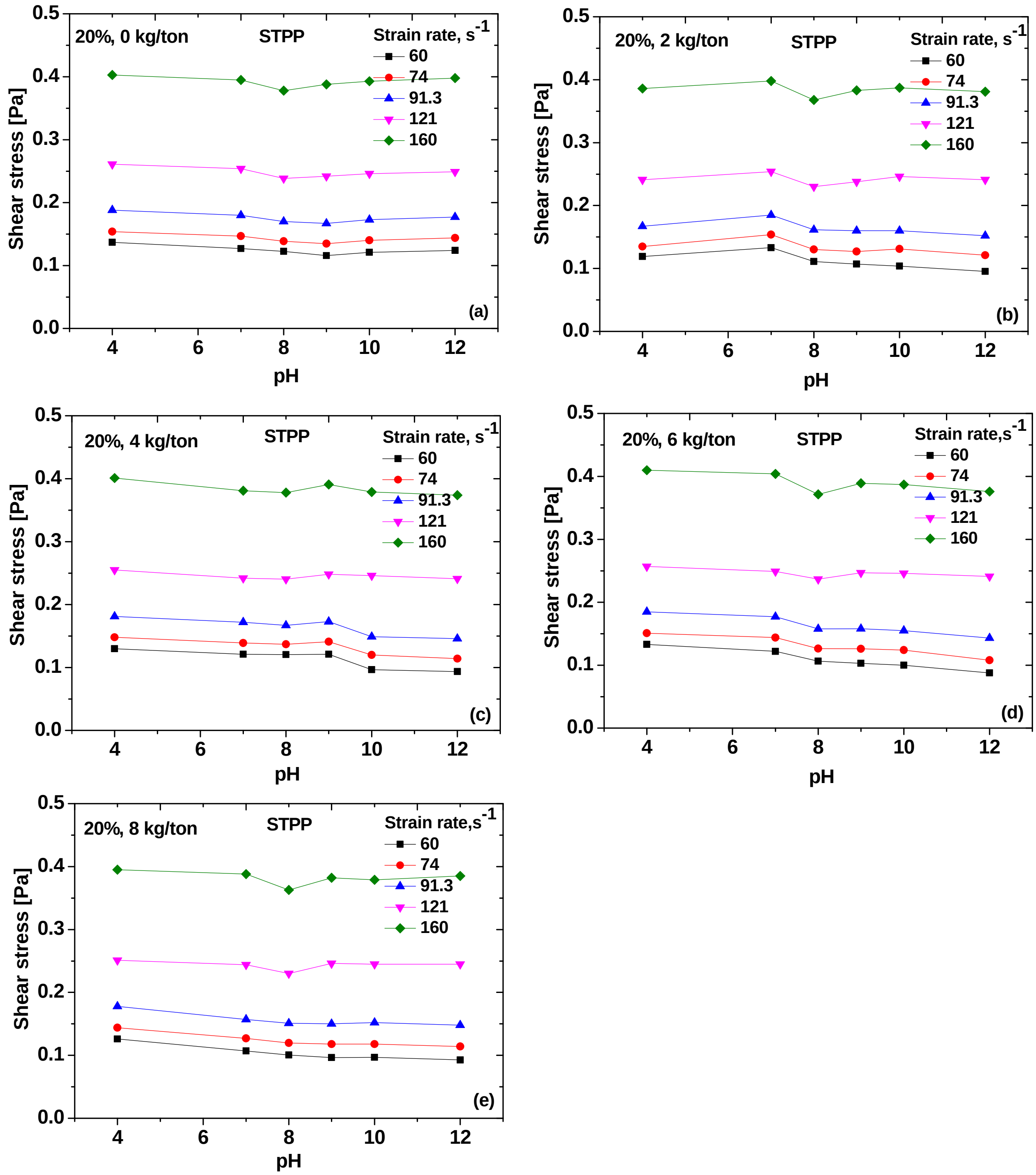


Figure 28. Effect of pH on CWS at 20% solids for different STPP dispersant dosages (a) 0.0 kg/ton, (b) 2.0 kg/ton, (c) 4.0 kg/ton, (d) 6.0 kg/ton and (e) 8.0 kg/ton.

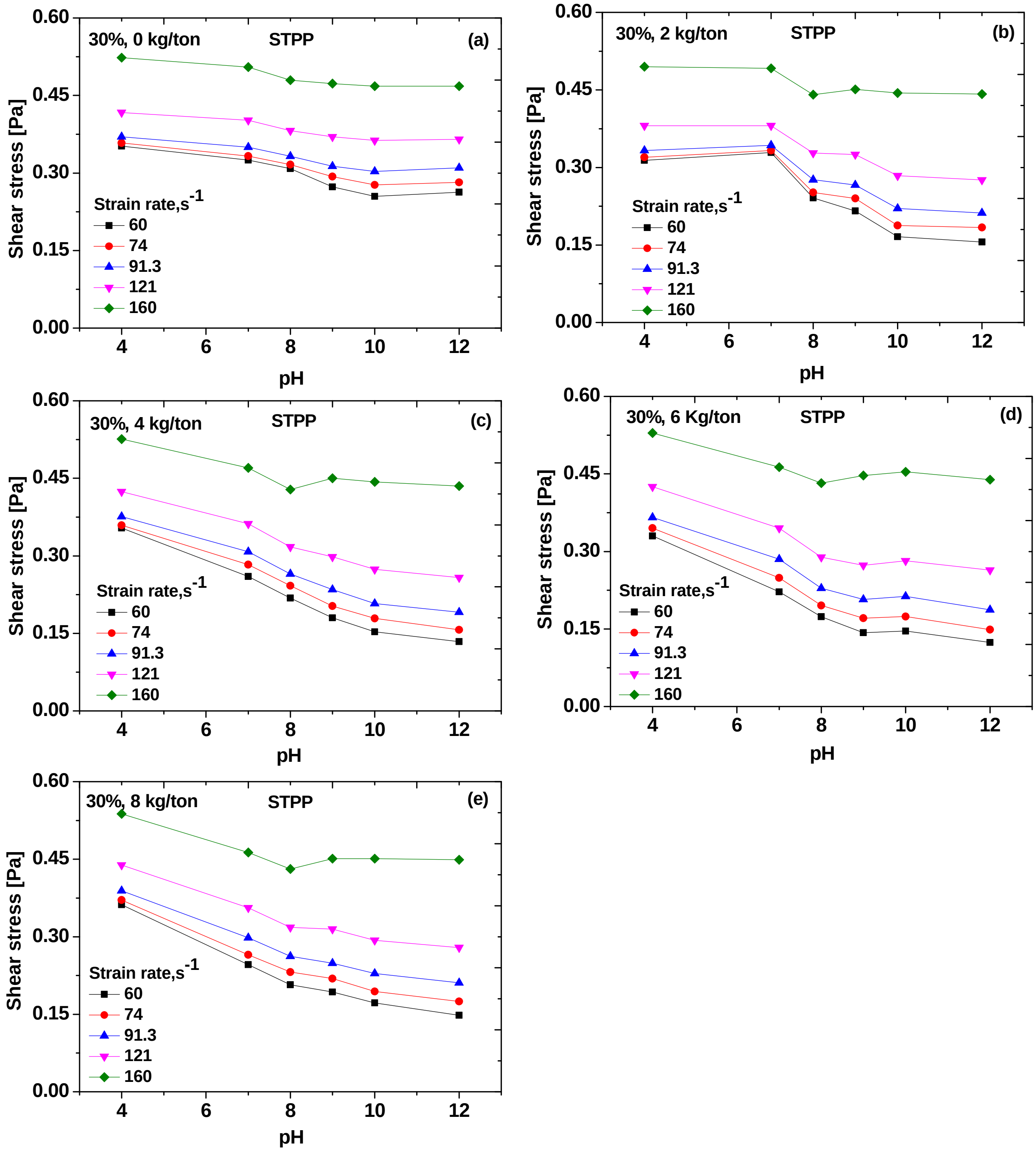


Figure 29. Effect of pH on CWS at 30% solids for different STPP dispersant dosages (a) 0.0 kg/ton, (b) 2.0 kg/ton, (c) 4.0 kg/ton, (d) 6.0 kg/ton and (e) 8.0 kg/ton.

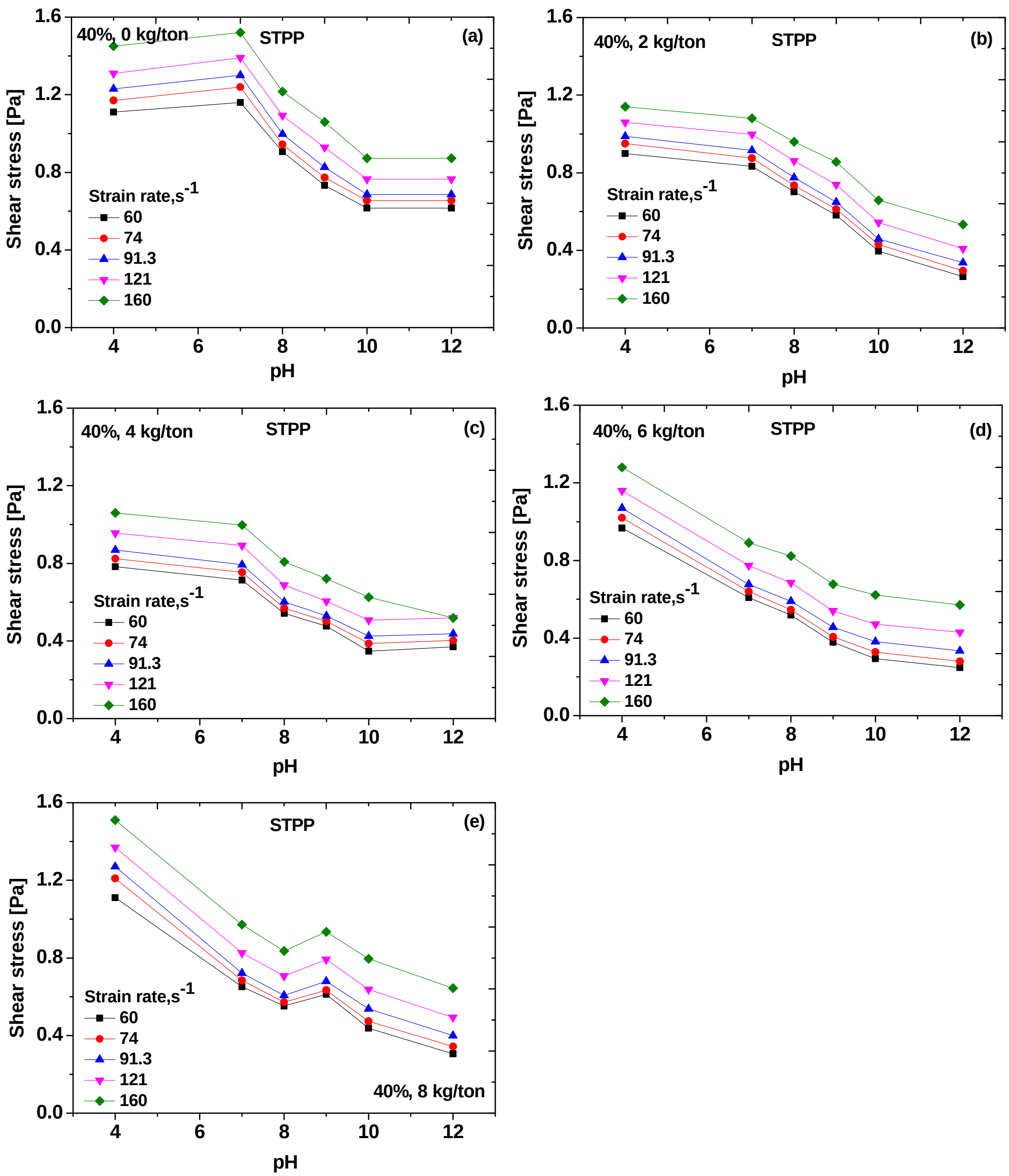


Figure 30. Effect of pH on CWS at 40% solids for different STPP dispersant dosages (a) 0.0 kg/ton, (b) 2.0 kg/ton, (c) 4.0 kg/ton, (d) 6.0 kg/ton and (e) 8.0 kg/ton.

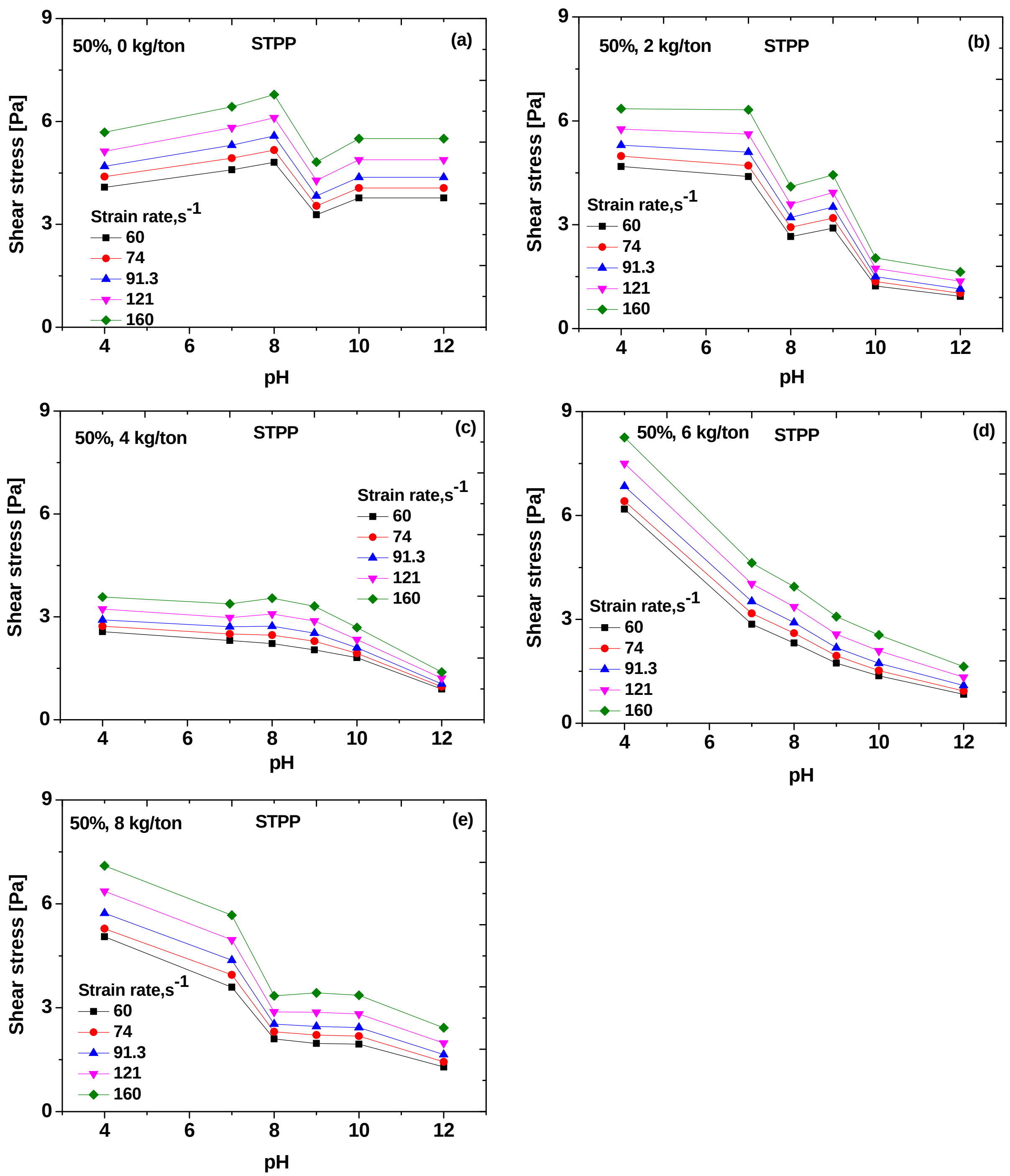


Figure 31. Effect of pH on CWS at 50% solids for different STPP dispersant dosages (a) 0.0 kg/ton, (b) 2.0 kg/ton, (c) 4.0 kg/ton, (d) 6.0 kg/ton and (e) 8.0 kg/ton.

4.1.5 Effect of flow behaviour index on CWS for STPP as dispersant

Fig.32 (a-e) shows the flow behaviour index values are plotted for all CWS with respect to solids concentration, dispersant dosage and pH. For 10% CWS, the effect of pH on flow behaviour index is not much pronounced as the values are very close in magnitude and the slurries are dilatant in nature. For 20%-50%, the effect of pH on flow behaviour index is noticeable. For a given solids concentration, the flow behaviour index is increasing with increase in pH in many cases. Generally, shear thinning behaviour is favourable for the transportation of slurry owing to the decrease in the viscosity with the increase of shear rate. Interestingly, CWS of 10% and 20% solid loadings exhibited shear thickening behaviour for all dispersant dosages and pH values tested while 40% and 50% are shear thinning in nature. CWS of 30% solid loading displayed shear thickening behaviour at higher solid loadings and higher pH values.

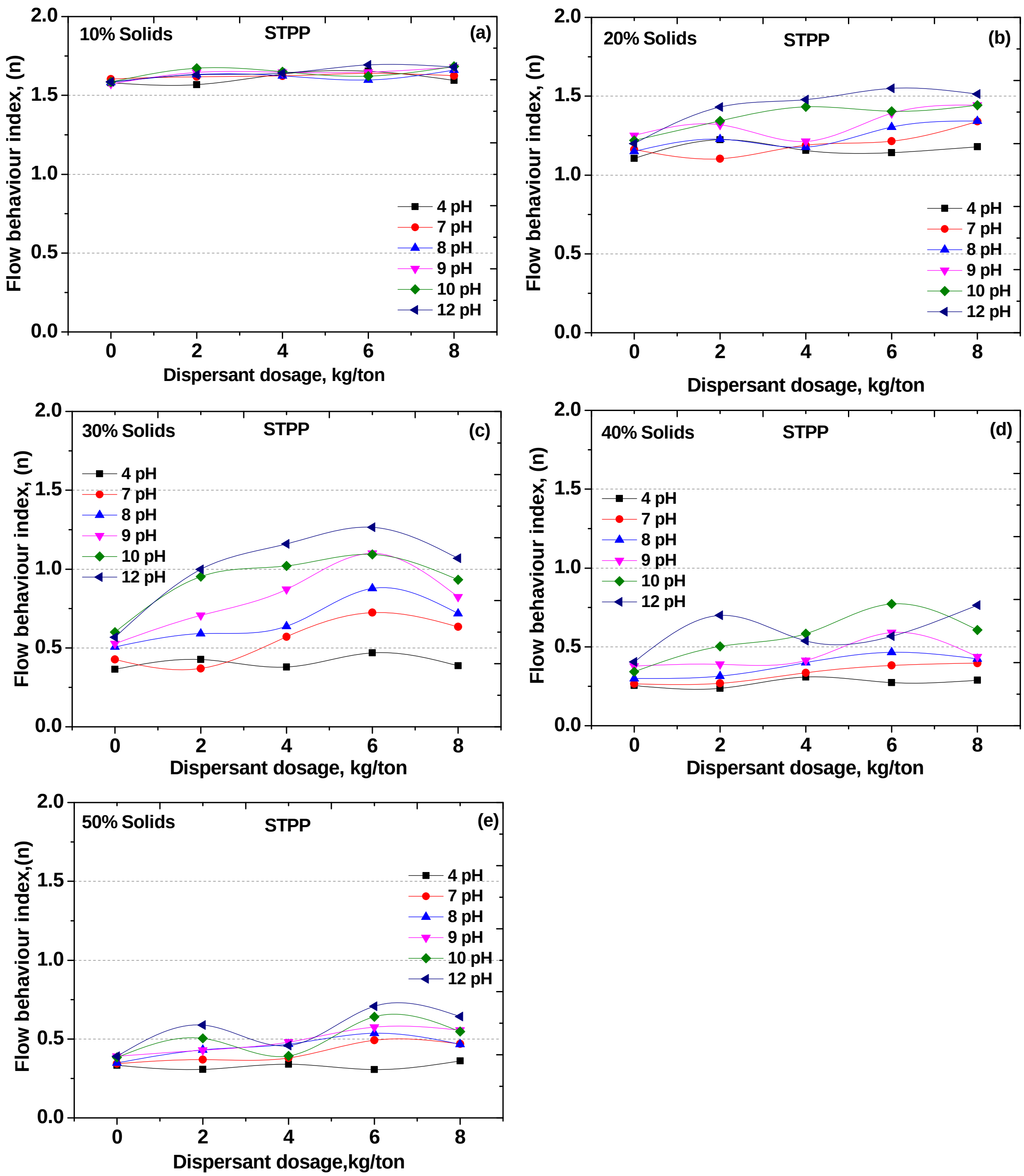


Figure 32. Effect of STPP dosage on flow behaviour index (n) for (a) 10, (b) 20, (c) 30, (d) 40 and (e) 50 percent solids at different pH.

4.1.6 Summary

At lower shear rates, the rheological nature of CWS is investigated. The role of Sodiumtripolyphosphate (STTP) in favourable alteration of the rheological nature of the slurry for the coal variety indicates the suitability of dispersant for the coal in slurry transport application. The effect of dispersant at 4 and 6 kg /ton loading is clearly evident in reducing the shear stress values for 30 %, 40% and 50% solid loading for all pH values. Interestingly, the shear stress- shear rate relation did not alter with respect to dispersant dosage or pH at lower solid loadings (10 %, 20 %) and they exhibited shear thickening nature in the predictions based on power law model. The slurry with 30% solid loading showed transition from shear thickening to shear thinning behaviour with the increase in dispersant dosage and pH. For the higher solid loadings, the slurry exhibited shear thinning or pseudoplastic behaviour at higher pH values with the addition of dispersant. The addition of anionic dispersant (STTP) at higher pH has yielded to favourable pumpable characteristics.

4.2 Rheological behaviour of CWS with Carboxymethylcellulose (CMC) as dispersant

4.2.1 Introduction

The flow characteristics of the coal water slurries depends on (1) physical and chemical properties of the coal such as ash content, the amount of inherent water, the degree of coal oxidation, and the quantity of surface active functional groups;(2) the volume fraction, ϕ, of the suspension; (3) the particle size range and its distribution, (4) interparticle interactions in the suspension and their effects of pH and the chemical additives etc. Chemical additives are important ingredients in reducing the viscosity, maintaining fluidity and improving the stability of CWS by introducing the electrostatic or steric repulsions or increasing the steric wettability of coal.

This chapter describes the rheological behaviour of CWS in the presence of an anionic polymeric dispersant namely carboxymethylcellulose (CMC, Chemical Formula: $C_8H_{15}NaO_8$) using Indian low rank coal variety (*Coal 1)* mined in Jamadoda, Jharkhand state. For all CWS, the effect of different solids loading (10%, 20%, 30%, 40% and 50%), dispersant dosages (0.5, 1.0, 1.5 and 2.0 kg/ton) and slurry pH (4,7, 8, 9, 10 and 12) on flow behaviour is studied at shear rate in the range of 60-160 s^{-1}. The rheology data was fitted for power law model (refer equation (1) as shown in Chapter 2, Section 2.1.2.1) and the flow behaviour index was estimated to identify the rheological nature as a function of solids loading, dispersant dosage and pH of the of the slurry.

4.2.2 Effect of solids concentration on CWS for CMC as dispersant

The rheological data plotted as the variation shear stress with respect to dispersant dosage (constant shear rate curves) are presented in Fig.33 (a-f) – Fig.37 (a-f). Upon closer examination of the plots, the rheological behaviour of all suspensions at different pH values is found to be Non-Newtonian.

For the lower percentage of solids (10%, 20%), the shear stress versus shear rate relations are almost similar in nature and magnitude with respect to dispersant dosage for all pH values (Fig.33 (a-f) – Fig.34 (a-f)). On the other hand, the dispersant effect is much pronounced for the higher solid loadings (30%, 40% and 50%). For the increase in solids concentration, an increase in the shear stress values is seen (Fig.35 (a-f) – Fig.37 (a-f)). Due to the shear interaction of particles and significant friction among them can be seen as the reason for the same [11,60].

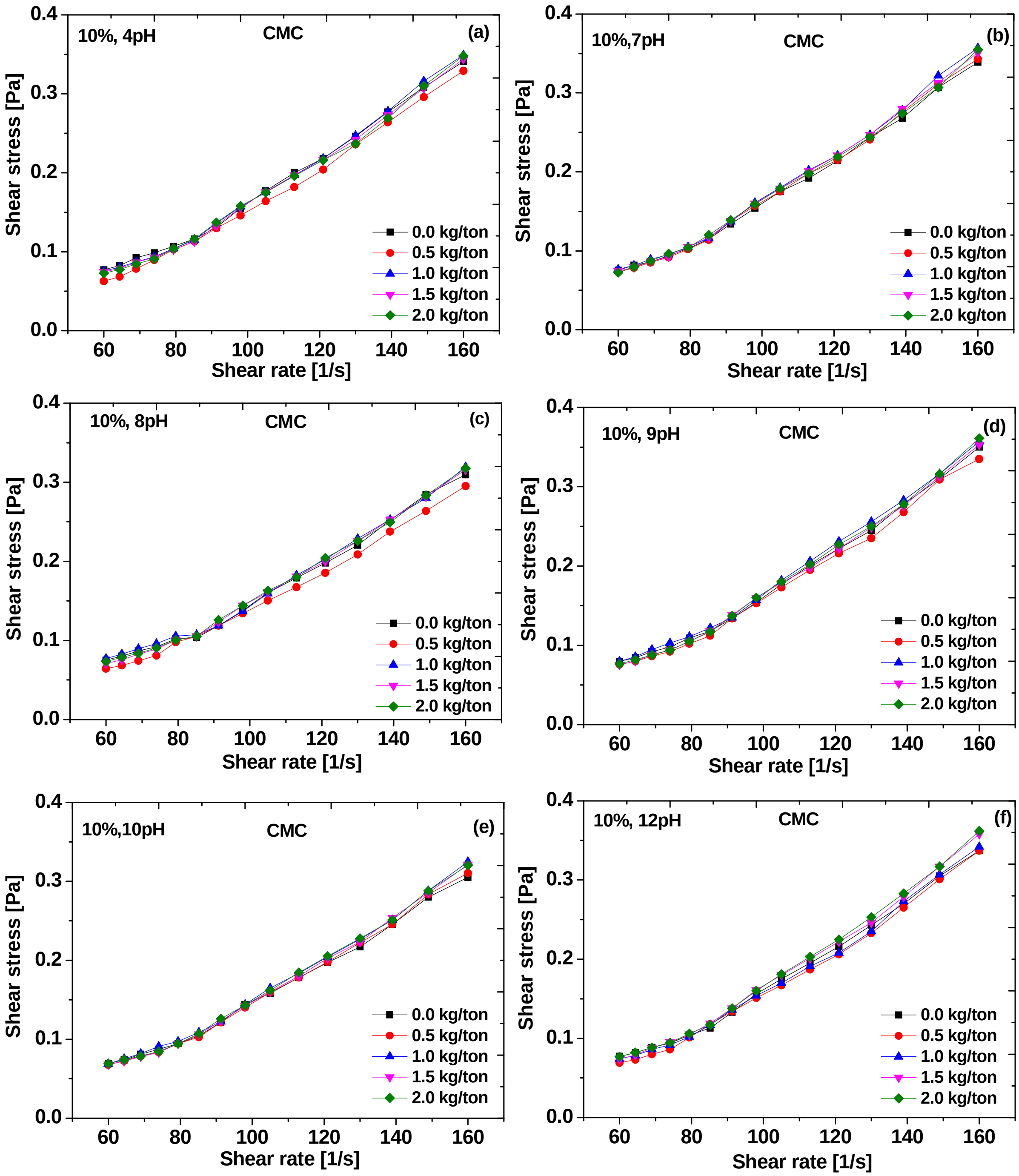


Figure 33. Shear stress versus shear rate on CWS of 10% solids concentration at pH (a) 4, (b)7, (c) 8, (d) 9, (e) 10 and (f) 12 for CMC as dispersant

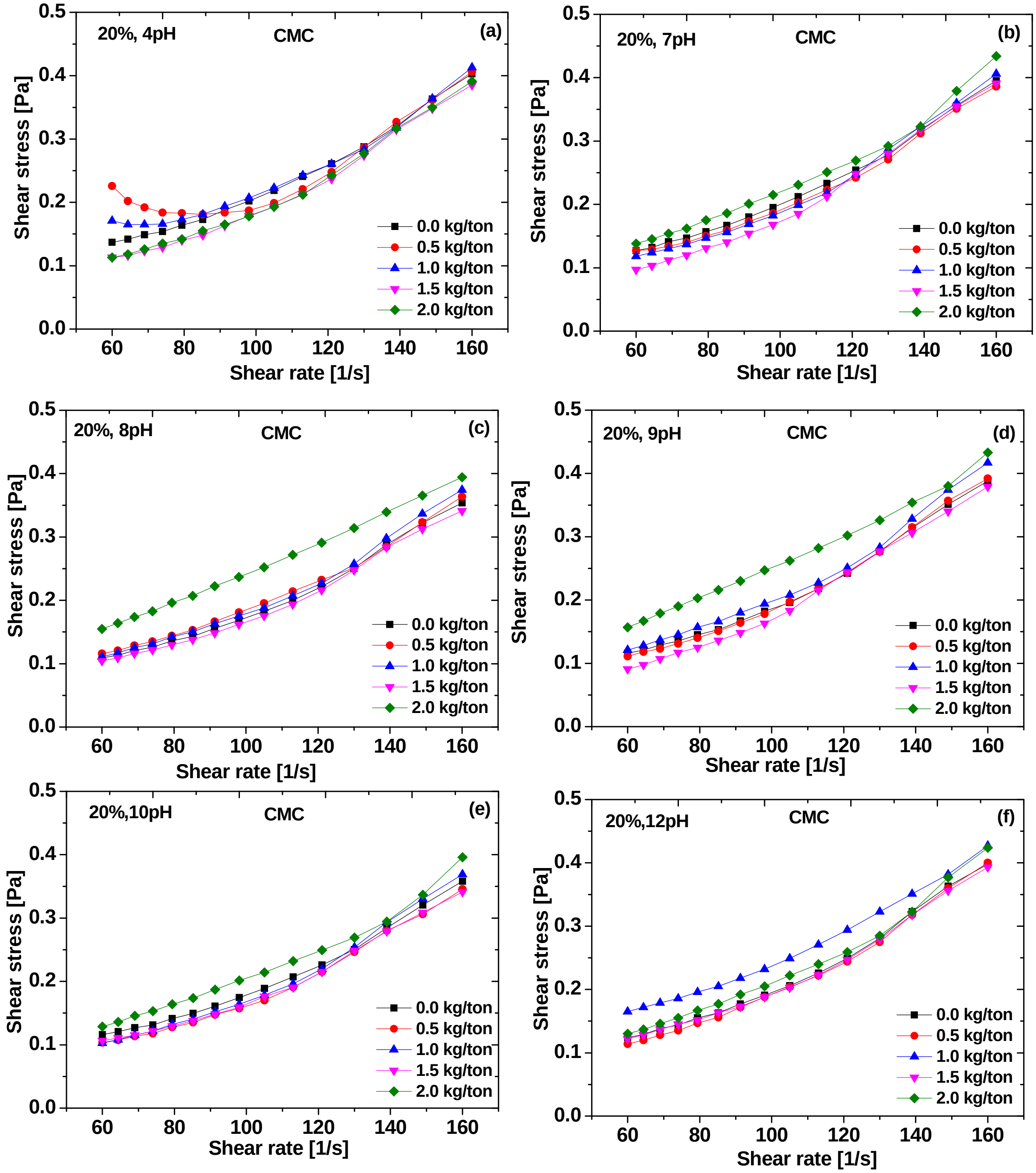


Figure 34. Shear stress versus shear rate on CWS of 20% solids concentration at pH (a) 4, (b)7, (c) 8, (d) 9, (e) 10 and (f) 12 for CMC as dispersant

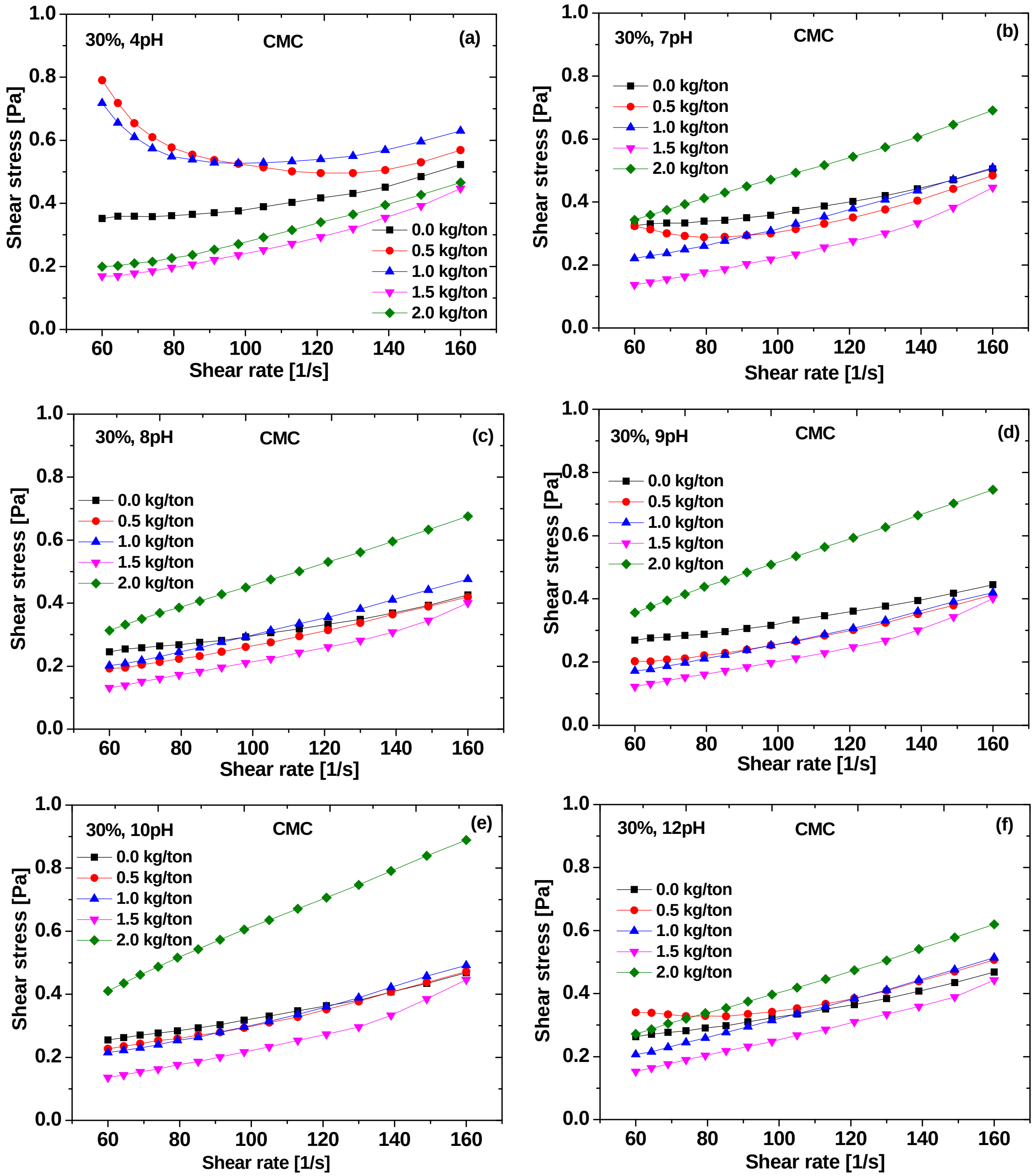


Figure 35. Shear stress versus shear rate on CWS of 30% solids concentration at pH (a) 4, (b)7, (c) 8, (d) 9, (e) 10 and (f) 12 for CMC as dispersant.

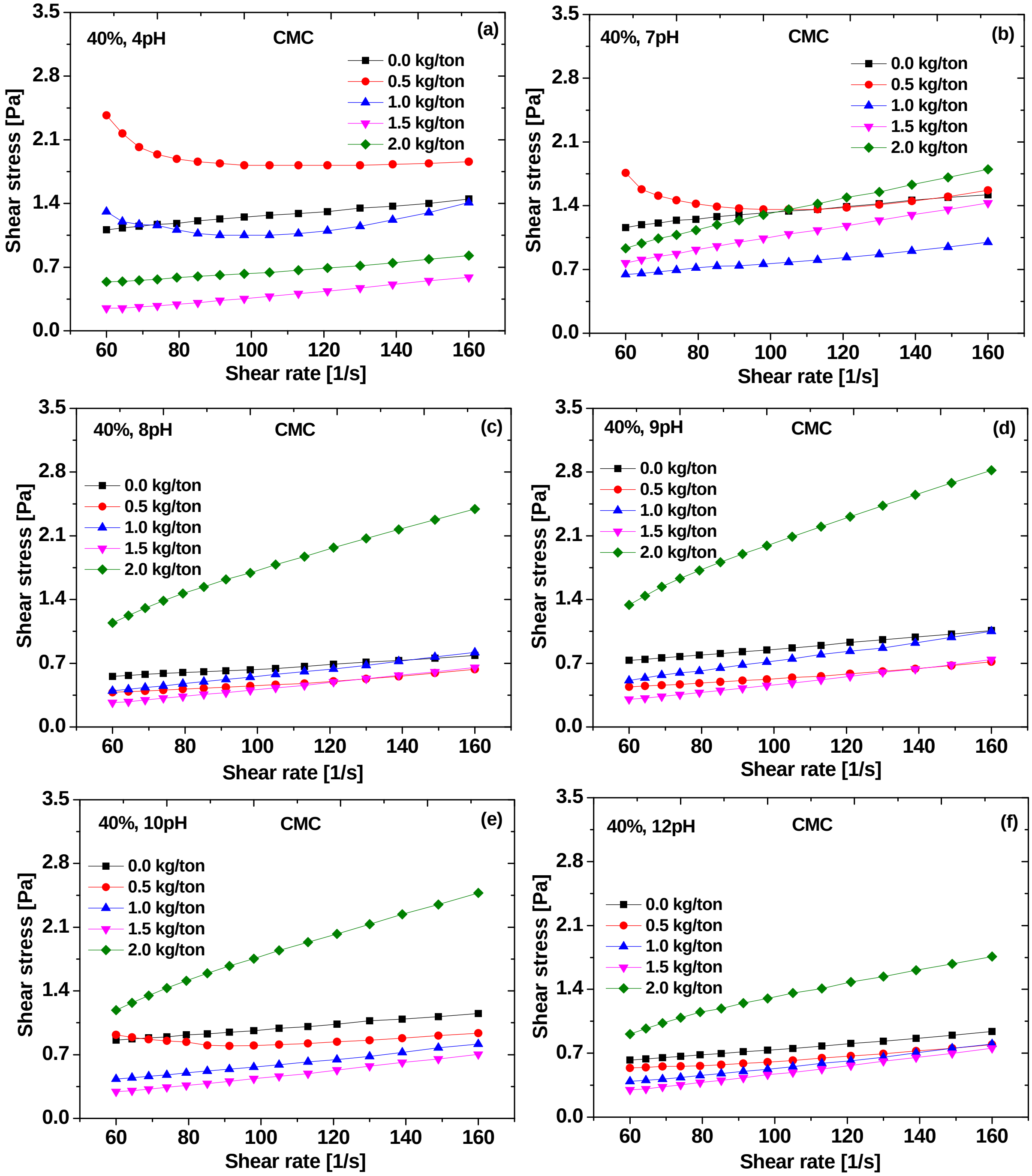


Figure 36. Shear stress versus shear rate on CWS of 40% solids concentration at pH (a) 4, (b)7, (c) 8, (d) 9, (e) 10 and (f) 12 for CMC as dispersant

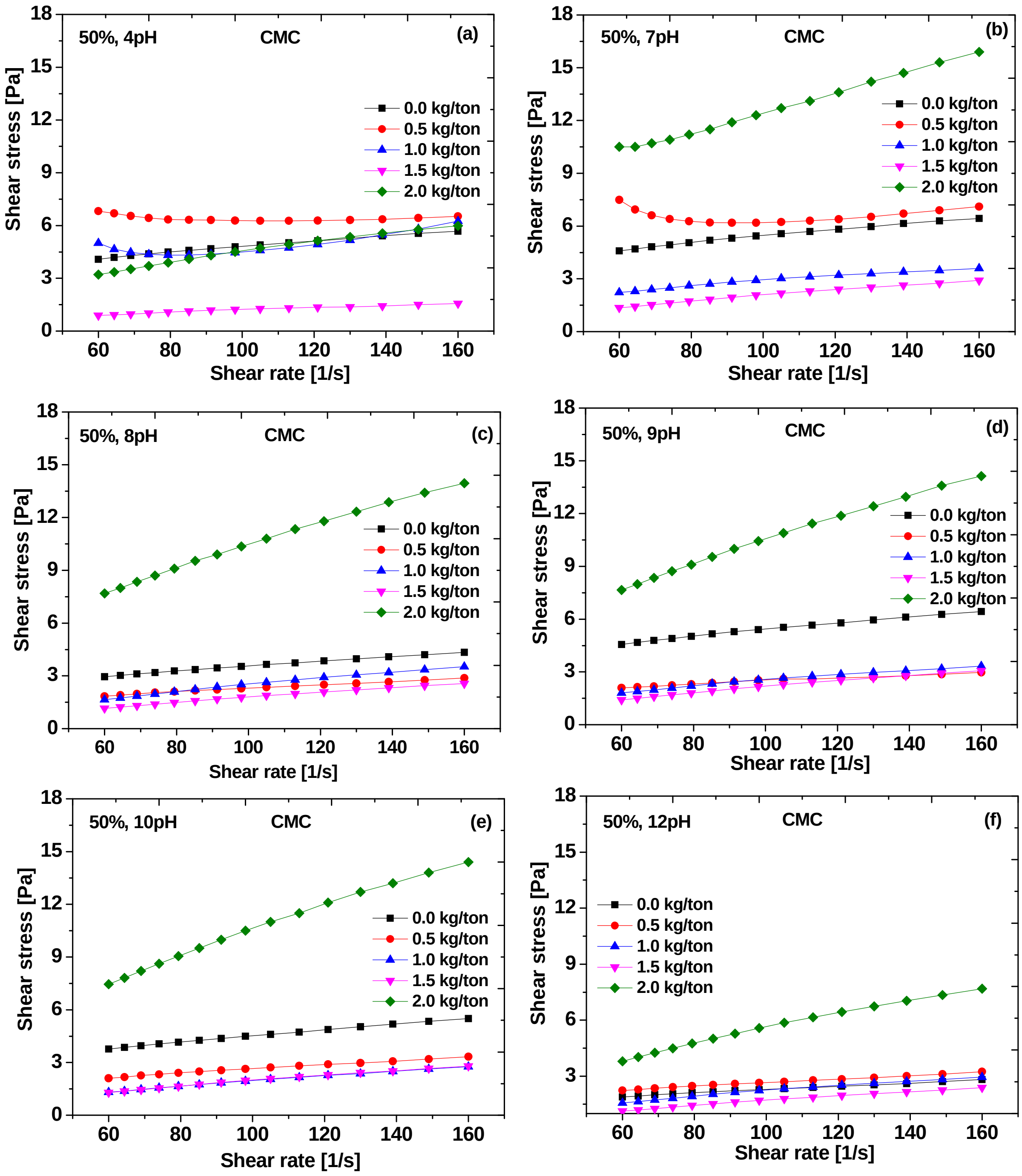


Figure 37. Shear stress versus shear rate on CWS of 50% solids concentration at pH (a) 4, (b)7, (c) 8, (d) 9, (e) 10 and (f) 12 for CMC as dispersant

4.2.3 Effect of dispersant on CWS for CMC as dispersant

To understand the effect of dispersant dosage on shear rate values, the rheological data plotted as the variation shear stress with respect to dispersant dosage (constant shear rate curves) are presented in Fig.38 (a-f) - Fig 42 (a-f). The dispersant dosage has no significant effect on the shear stress values for 10% and 20% solid loading for all pH values as observed in Fig.38 (a-f) - Fig.39 (a-f). For 30% solid loading (Fig.40 (a-f)), for a given pH, the shear stress attains minima at dispersant dosage of 1.5 kg /ton for all shear rates tested. For the highest shear rate tested (160 s^{-1}), the dispersant has marginal effect as the magnitude of shear stress change is minimal for this solid loading.

In the case of 40% solid loading (Fig.41 (a-f)), for a given pH, the minimum shear stress attains minima at dispersant dosage 1.5 kg/ton, except for pH 7. A minimum shear stress is reported at dispersant dosage of 1.0 kg/ton for a pH of 7. For the highest shear rate tested (160 s^{-1}), the dispersant has marginal effect as the magnitude of shear stress change is minimal for this solid loading for pH 12. Two local minima values are seen in the case of pH 9 and pH 10, Minimum shear stress values are seen for all shear rate values at dispersant dosage 0.5 kg/ ton and 1.5 kg/ton respectively. At higher shear rate tested ((121 s^{-1} and 160 s^{-1}), the minimum shear rate is reported at 0.5 kg/ton and 1.5 kg/ton.

For 50% solid loading and for a given pH (Fig.42 (a-f)), the minimum shear stress values obtained at 1.5 kg per ton of dispersant dosage except for pH 10. The minimum shear stress is obtained at a dispersant dosage between 1.0 kg/ton to 1.5 kg/ton at pH 10. For higher shear rate tested (121 s^{-1} and 160 s^{-1}) the dispersant has marginal effect as the magnitude and reported minimum shear stress at dosage of 1.5 kg/ton and 0.5 kg/ton for pH of 8 and 9 respectively. Two local minima of shear stress are reported at a dosage of 0.5 kg/ton and 1.5 kg/ton at pH of 8 and 9. Where as in the case of pH 10, two local minima in shear stress is reported at a dispersant dosage of 1.0 kg/ton and 1.5 kg/ton.

For a given percent solids and pH, an increase of shear stress was observed for dispersant dosage between 1.5 kg/ ton to 2.0 kg/ton. This can be due to agglomeration of particles in the slurry after attaining saturation limit of the dispersant. The excessive dispersant dosage expected to increase the ionic strength of the slurry which can result in formation of strong electrical double layer around the solid particles and thereby obvious reduction in the electrostatic repulsive forces among the particles. As a result, the shear stress values increase with increase of dispersant loading after attaining the saturation limit [52,54]. For a given solids concentration and dispersant dosage, a decrease in shear stress is seen at higher shear rate values. This can be due to the continuous breakdown of slurry structure or continuous and sudden breakdown of aggregates in the slurry [53].

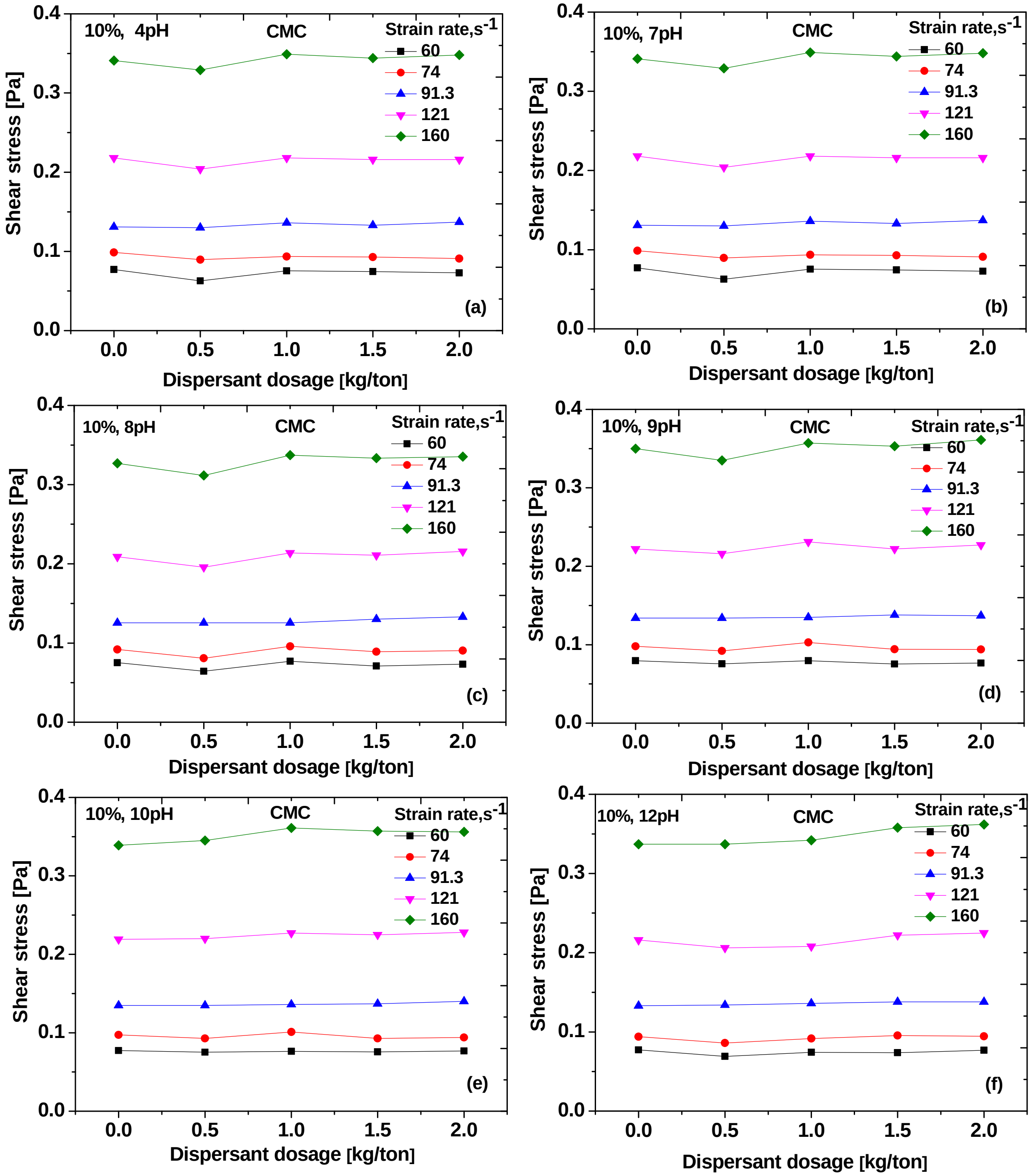


Figure 38. Effect of CMC dispersant dosage on 10% solid loadings at pH of (a) 4, (b) 7, (c) 8, (d) 9, (e) 10 and (f) 12.

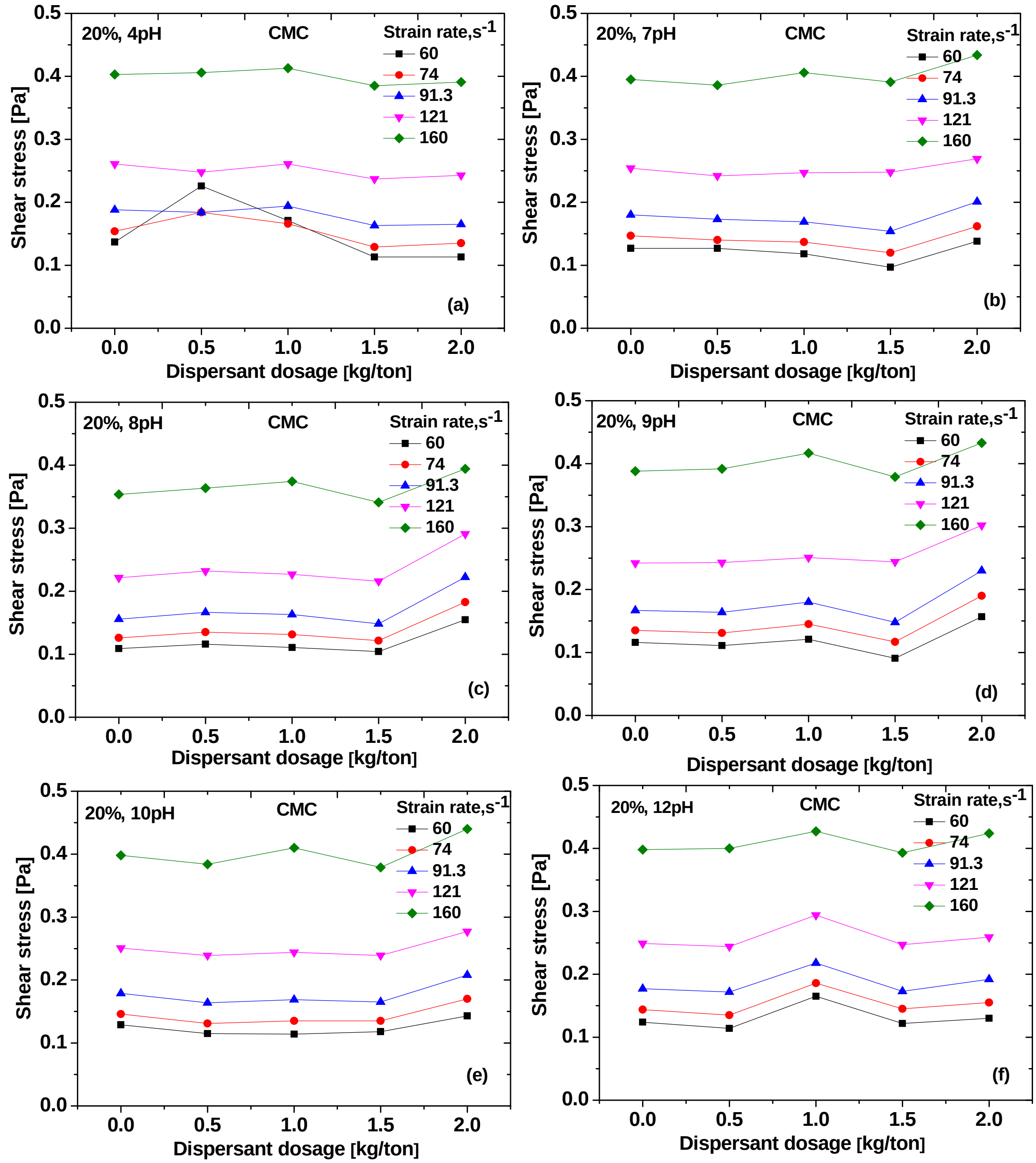


Figure 39. Effect of CMC dispersant dosage on 20% solid loadings at pH of (a) 4, (b) 7, (c) 8, (d) 9, (e) 10 and (f) 12.

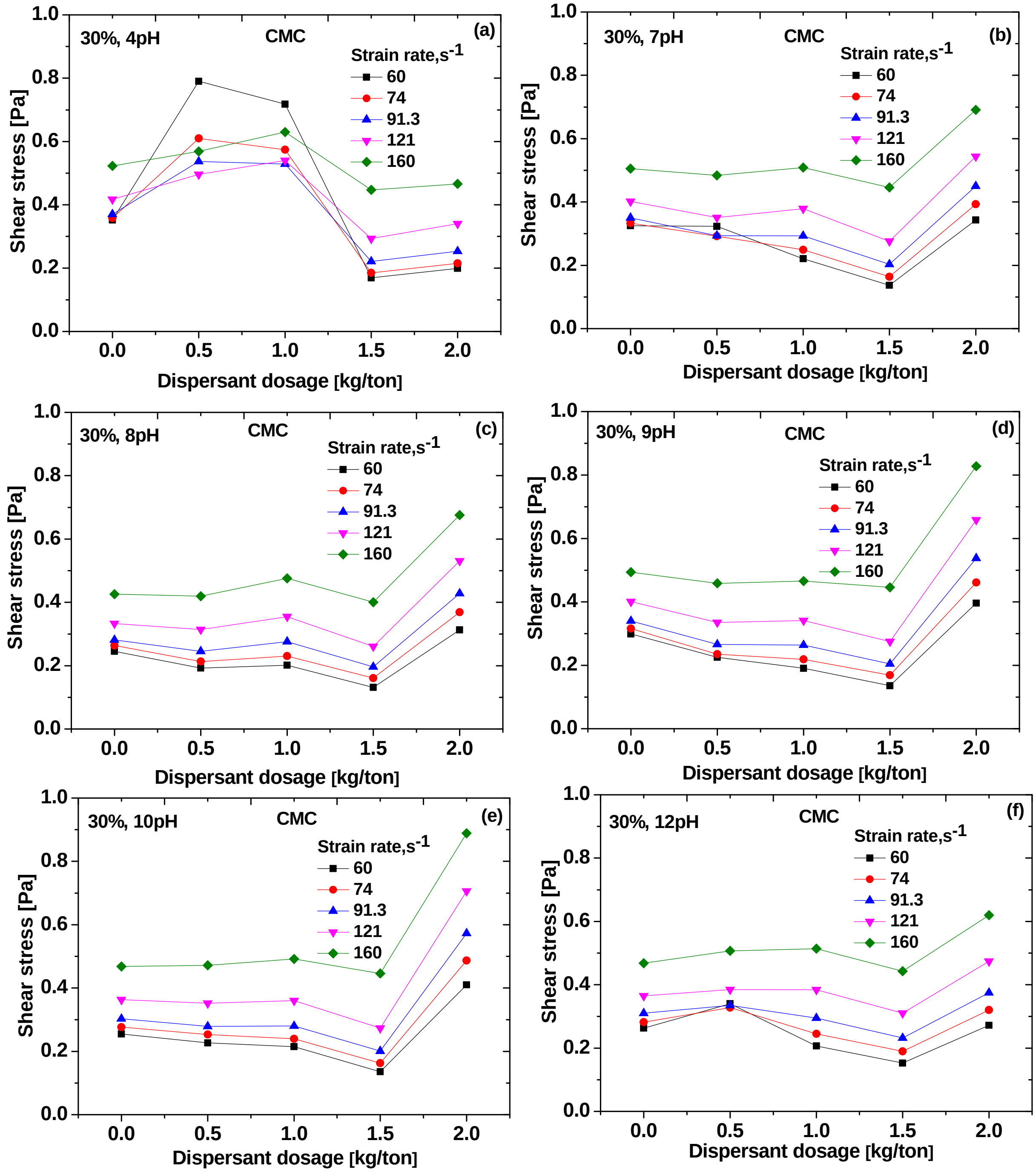


Figure 40. Effect of CMC dispersant dosage on 30% solid loadings at pH of (a) 4, (b) 7, (c) 8, (d) 9, (e) 10 and (f) 12.

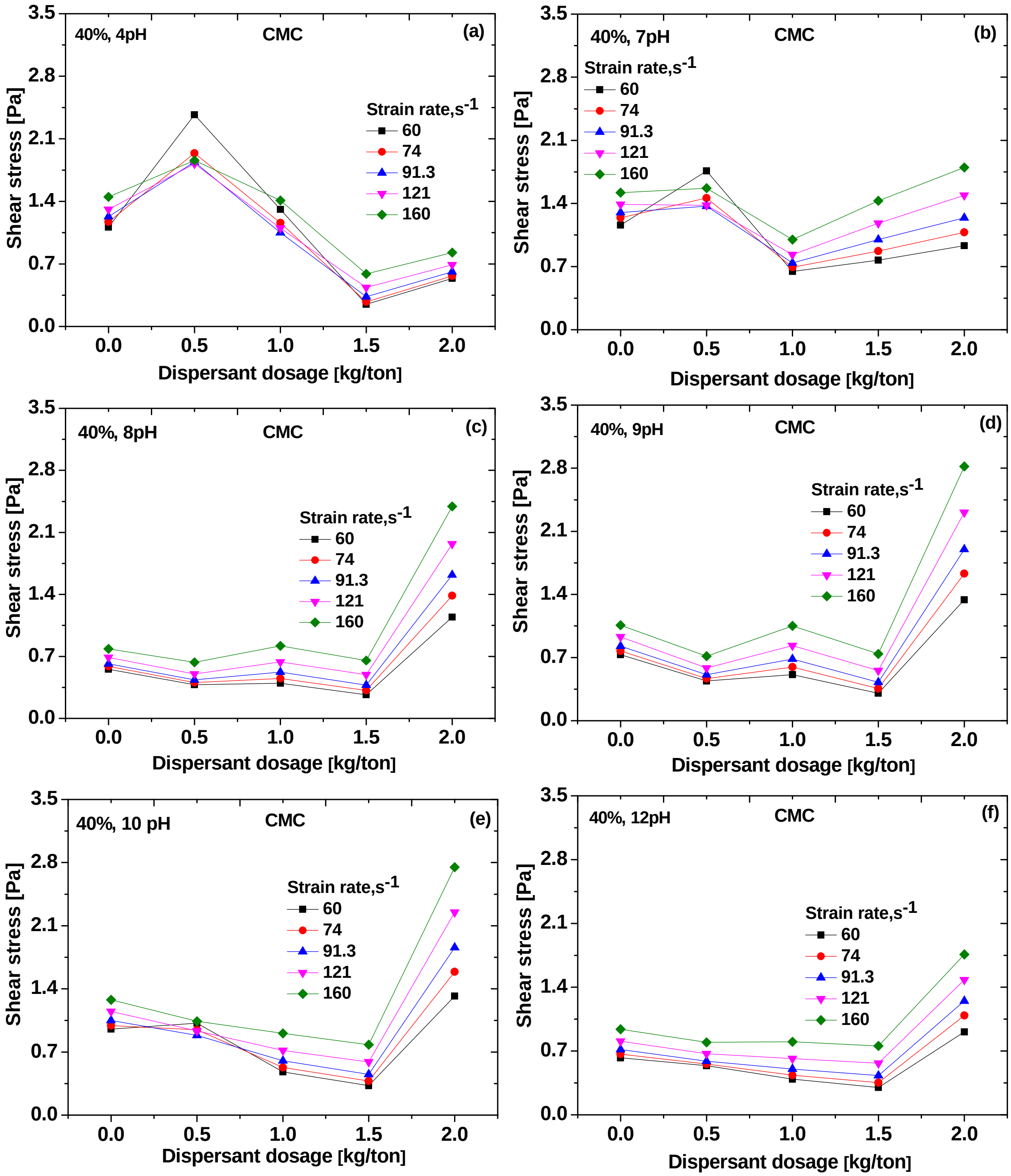


Figure 41. Effect of CMC dispersant dosage on 40% solid loadings at pH of (a) 4, (b) 7, (c) 8, (d) 9, (e) 10 and (f) 12.

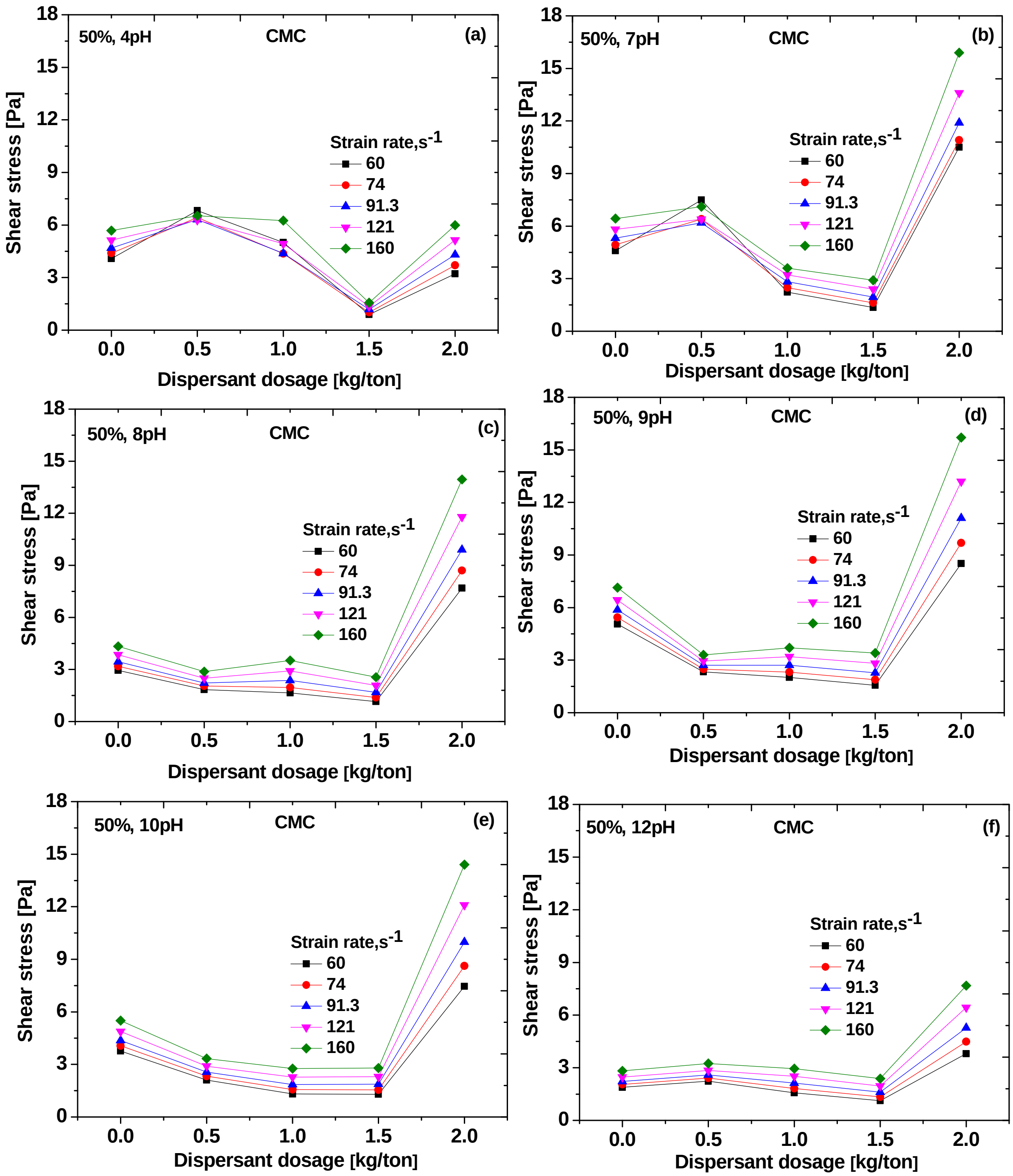


Figure 42. Effect of CMC dispersant dosage on 50% solid loadings at pH of (a) 4, (b) 7, (c) 8, (d) 9, (e) 10 and (f) 12.

4.2.4 Effect of pH on CWS for CMC as dispersant

To understand the effect of pH on shear rate values, the rheological data plotted as the variation shear stress with respect to pH (constant shear rate curves) are presented in Fig.43 (a-e) - Fig.47 (a-e). For 10% and 20% solids concentration, the pH has no significant effect on the shear stress for a given shear rate as observed in Fig.43 (a-e) - Fig.44 (a-e). For 30% solids loading (Fig.45 (a-e)), the shear stress values almost exhibited decreasing trend with an increase in pH for dispersant dosage up to 1.0 kg/ton. No significant change in shear stress with the pH is noticed for dispersant dosage of 1.5 kg/ton and 2.0 kg/ton. A drastic decrease of shear stress is observed for no dispersant slurry and at a dispersant dosage of 0.5 kg/ton.

For 40% loading (Fig.46 (a-e)), two local maximum shear stress values are attained at pH=9 at a dispersant dosage of 1.0 kg/ton and at pH -7 for dispersant dosage of 1.5 kg/ton for all the five shear rate values. At 50% solids concentration, the shear stress exhibited two local maximum values for the pH values of 8 and 10 with no dispersant and pH values of 9 and 8 for the dispersant dosages of 1.0 kg/ton and 1.5 kg/ton respectively, is shown in (Fig.47 (a-e)). The decrease of shear stress with respect to pH increase for all individual shear rates is evident for dispersant dosage up to 1.0 kg/ton. No significant change in shear stress with pH is noticed at dispersant dosage of 1.5 kg/ton. For the both the 40% and 50% solids, with the addition of dispersant dosage, the shear stress values exhibited decreasing trend with respect to pH in all the cases except for the dispersant dosage of 2.0 kg/ton. The surface chemistry of the suspension particles is crucial in attributing definite rheological properties to the slurry [26]. If the attractive forces among the particles are strong, higher shear forces are required to overcome the friction.

Overall, the effect of dispersant up to 1.5 kg per ton dosage is clearly evident in reducing the shear stress values for 30 %, 40% and 50% solid loading for all pH values as the adsorption of dispersant on the surface of the solids contributes to the countering of the attractive forces. At lower pH values, the presence of H^+ ions in the liquid media can hamper the adsorption of cations on the solid media surface. This phenomenon nullifies the effect of the dispersant. On the other hand, at higher pH values, the sufficient adsorption of cations on the particulate matter increases the electrostatic repulsions among them and contributes to the decrease in the shear stress values [55,56].

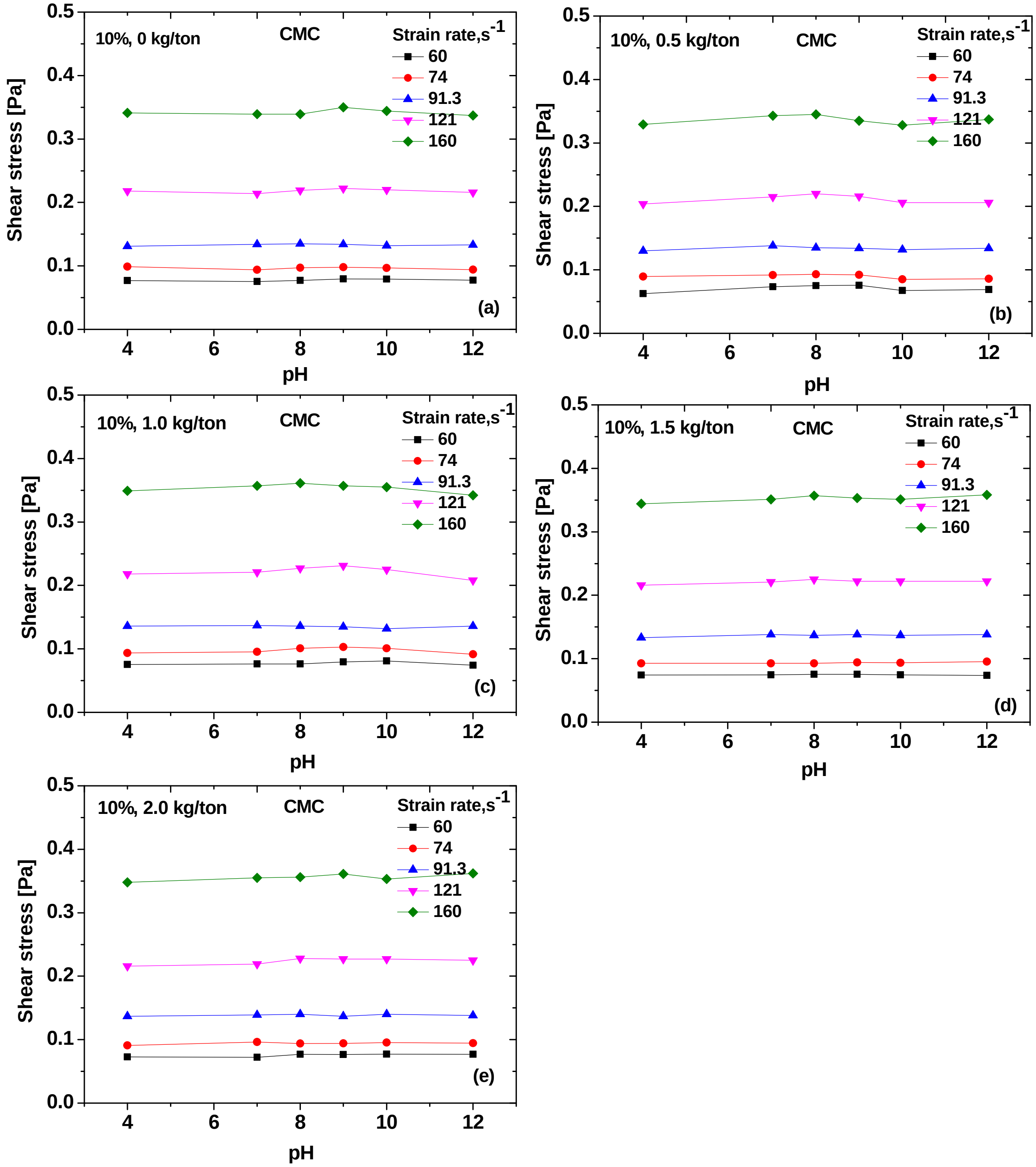


Figure 43. Effect of pH on CWS at 10% solids for different CMC dispersant dosages (a) 0.0 kg/ton, (b) 0.5 kg/ton, (c) 1.0 kg/ton, (d) 1.5 kg/ton and (e) 2.0 kg/ton.

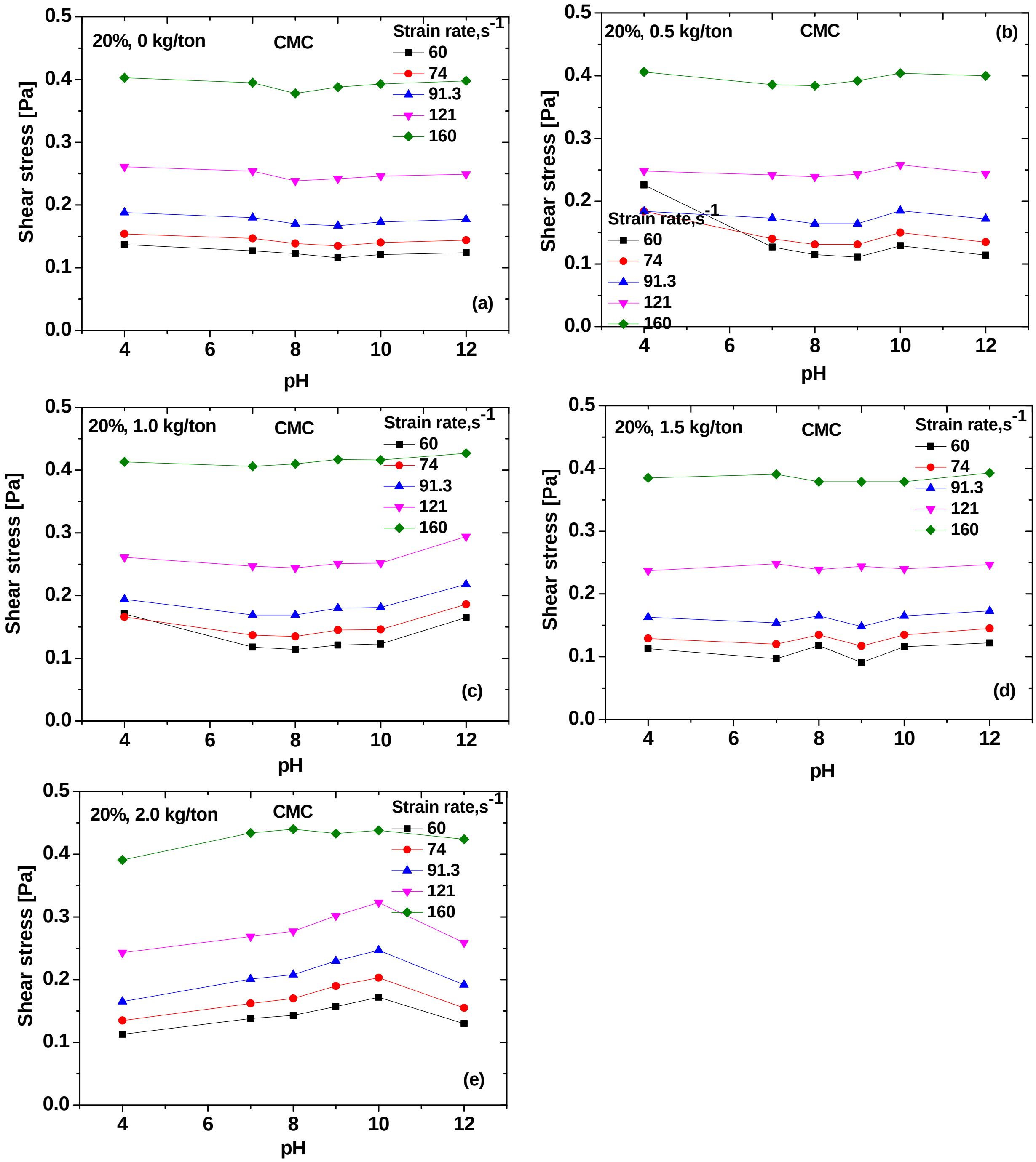


Figure 44. Effect of pH on CWS at 20% solids for different CMC dispersant dosages (a) 0.0 kg/ton, (b) 0.5 kg/ton, (c) 1.0 kg/ton, (d) 1.5 kg/ton and (e) 2.0 kg/ton.

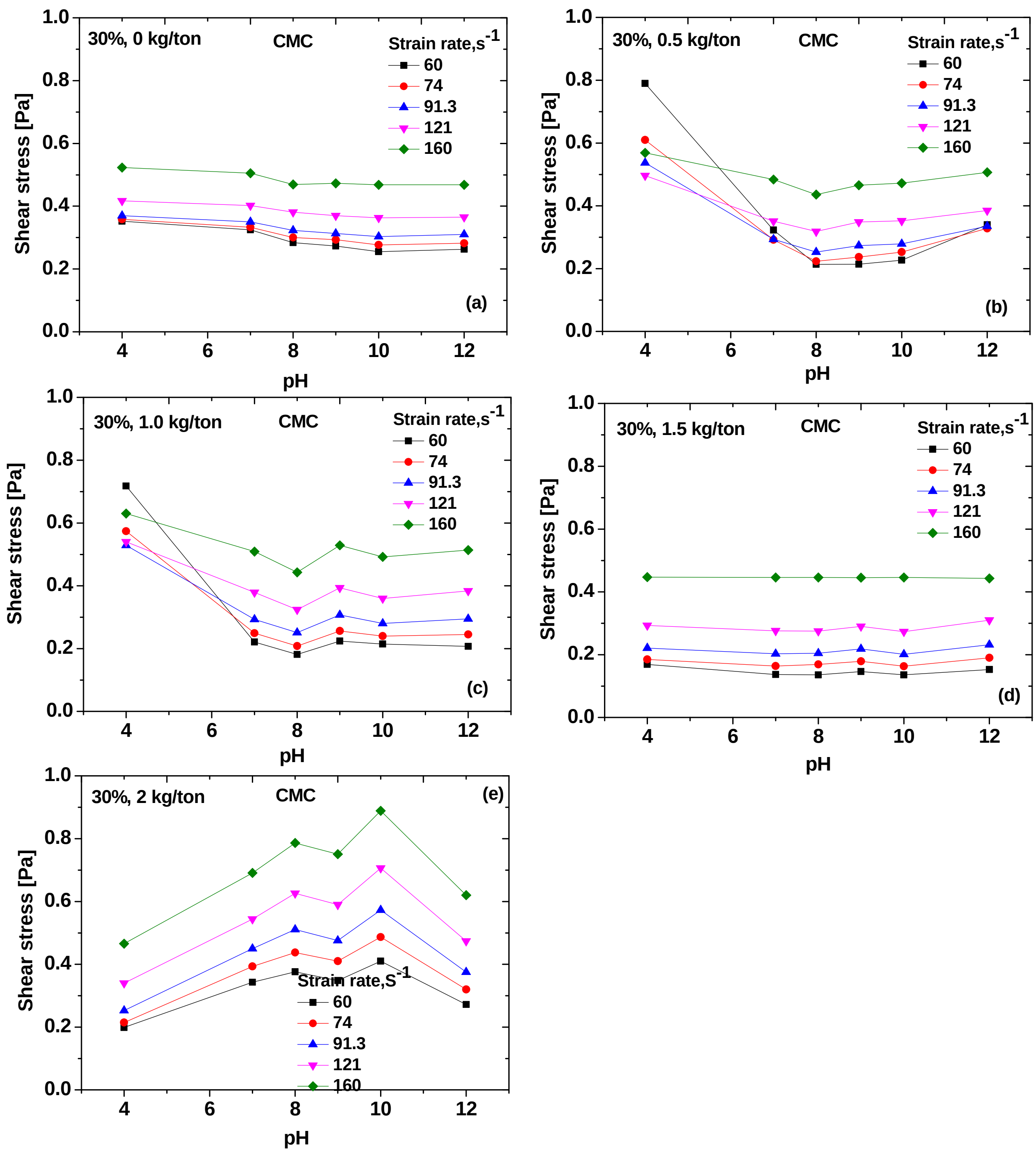


Figure 45. Effect of pH on CWS at 30% solids for different CMC dispersant dosages (a) 0.0 kg/ton, (b) 0.5 kg/ton, (c) 1.0 kg/ton, (d) 1.5 kg/ton and (e) 2.0 kg/ton.

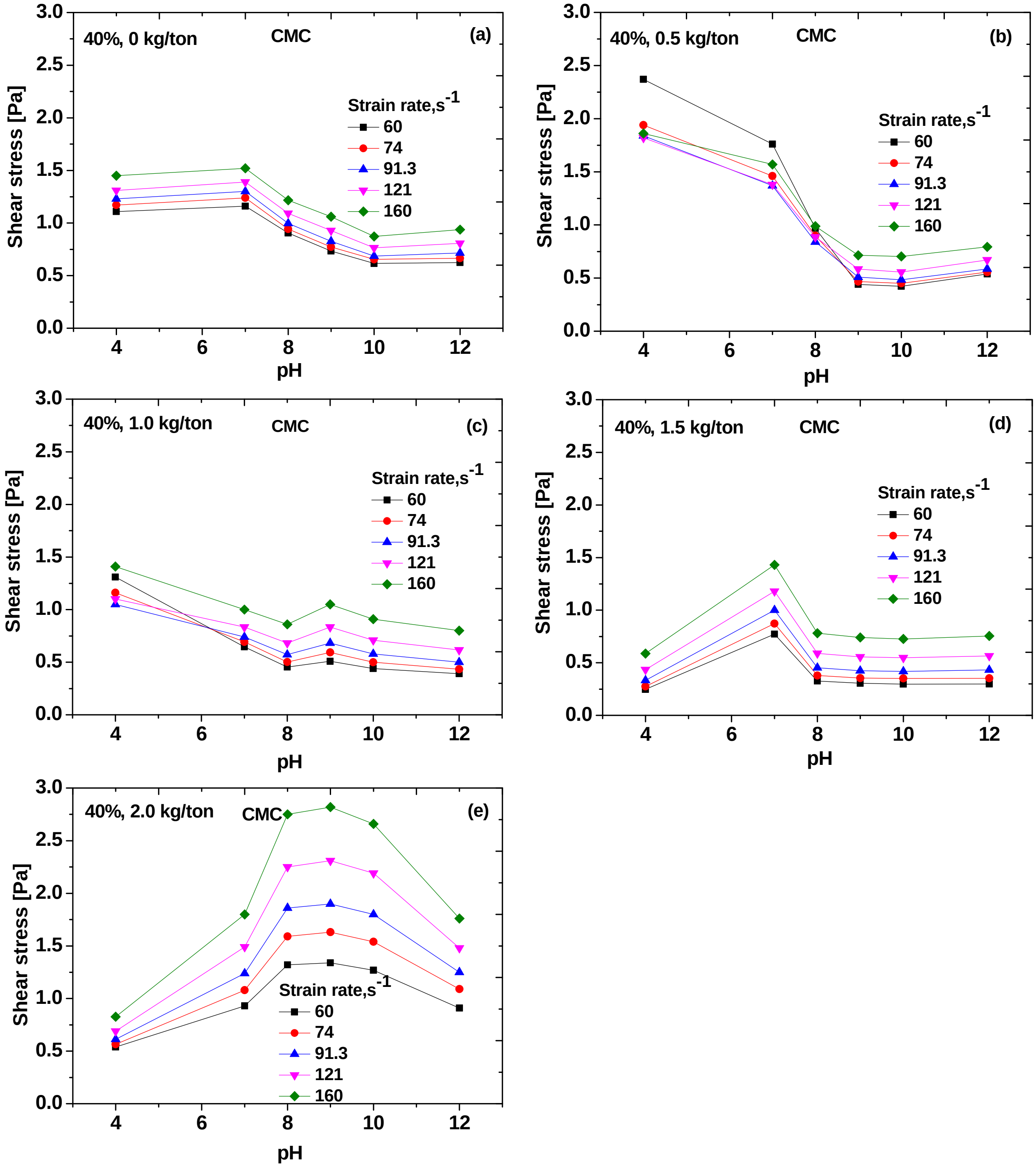


Figure 46. Effect of pH on CWS at 40% solids for different CMC dispersant dosages (a) 0.0 kg/ton, (b) 2.0 kg/ton, (c) 4.0 kg/ton, (d) 6.0 kg/ton and (e) 8.0 kg/ton.

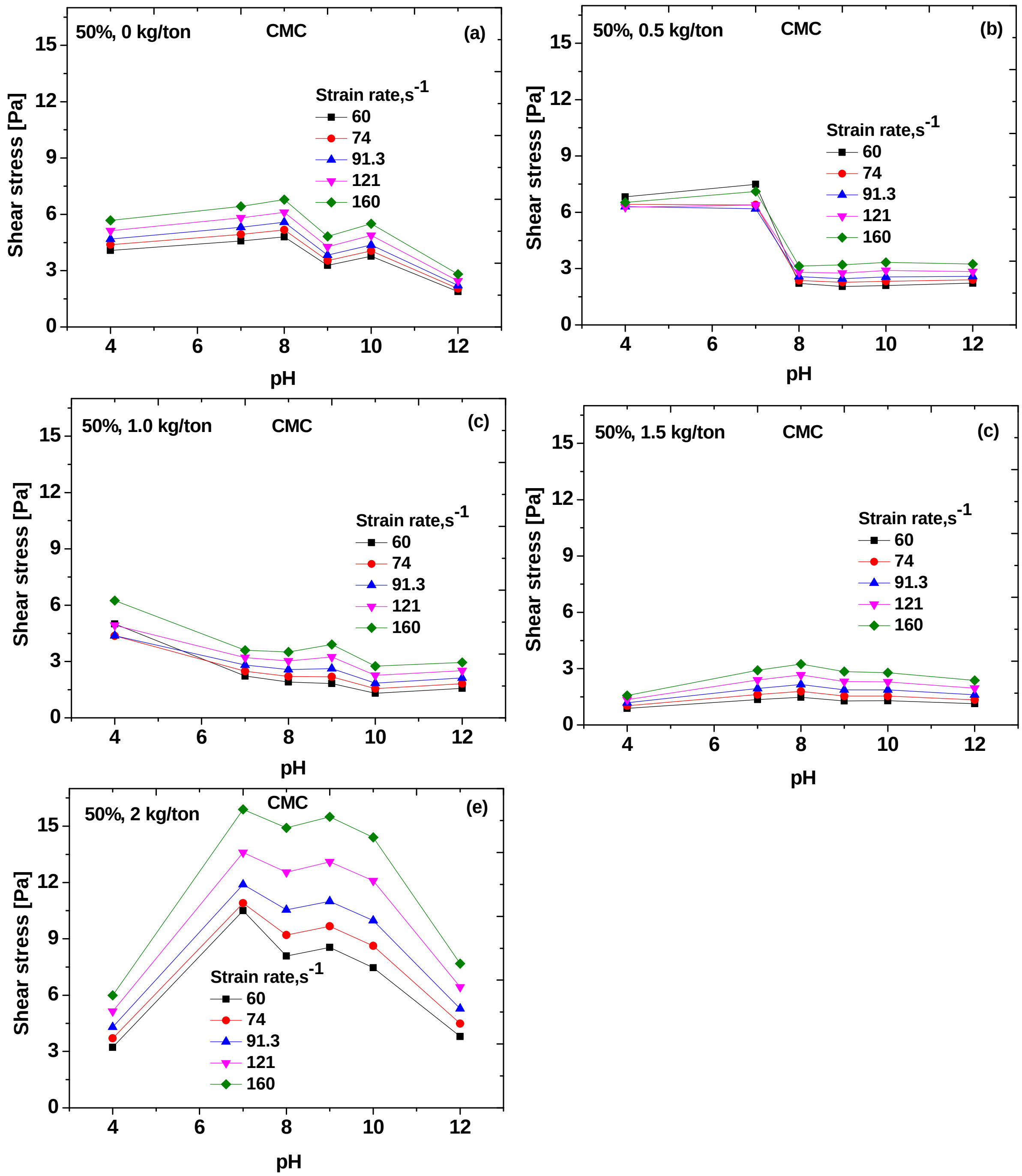


Figure 47. Effect of pH on CWS at 50% solids for different CMC dispersant dosages (a) 0.0 kg/ton, (b) 2.0 kg/ton, (c) 4.0 kg/ton, (d) 6.0 kg/ton and (e) 8.0 kg/ton.

4.2.5 Effect of flow behaviour index on CWS for CMC as dispersant

Fig.48 (a-e) shows the flow behaviour index values are plotted for all CWS with respect to solids concentration, dispersant dosage and pH. For 10% CWS, the effect of pH on flow behaviour index is not much pronounced as the values are very close in magnitude and the slurries are dilatant in nature. For 20%-50%, the effect of pH on flow behaviour index is noticeable. For a given solids concentration, the flow behaviour index is increasing with increase in pH in many cases. Generally, shear thinning behaviour is favourable for the transportation of slurry owing to the decrease in the viscosity with the increase of shear rate. Interestingly, CWS of 10% solid loadings exhibited shear thickening behaviour for all dispersant dosages and pH values tested while 40% and 50% are shear thinning in nature. The transition of flow behaviour from shear thickening and shear thinning is noticed at CWS of 20% and 30% solids concentration.

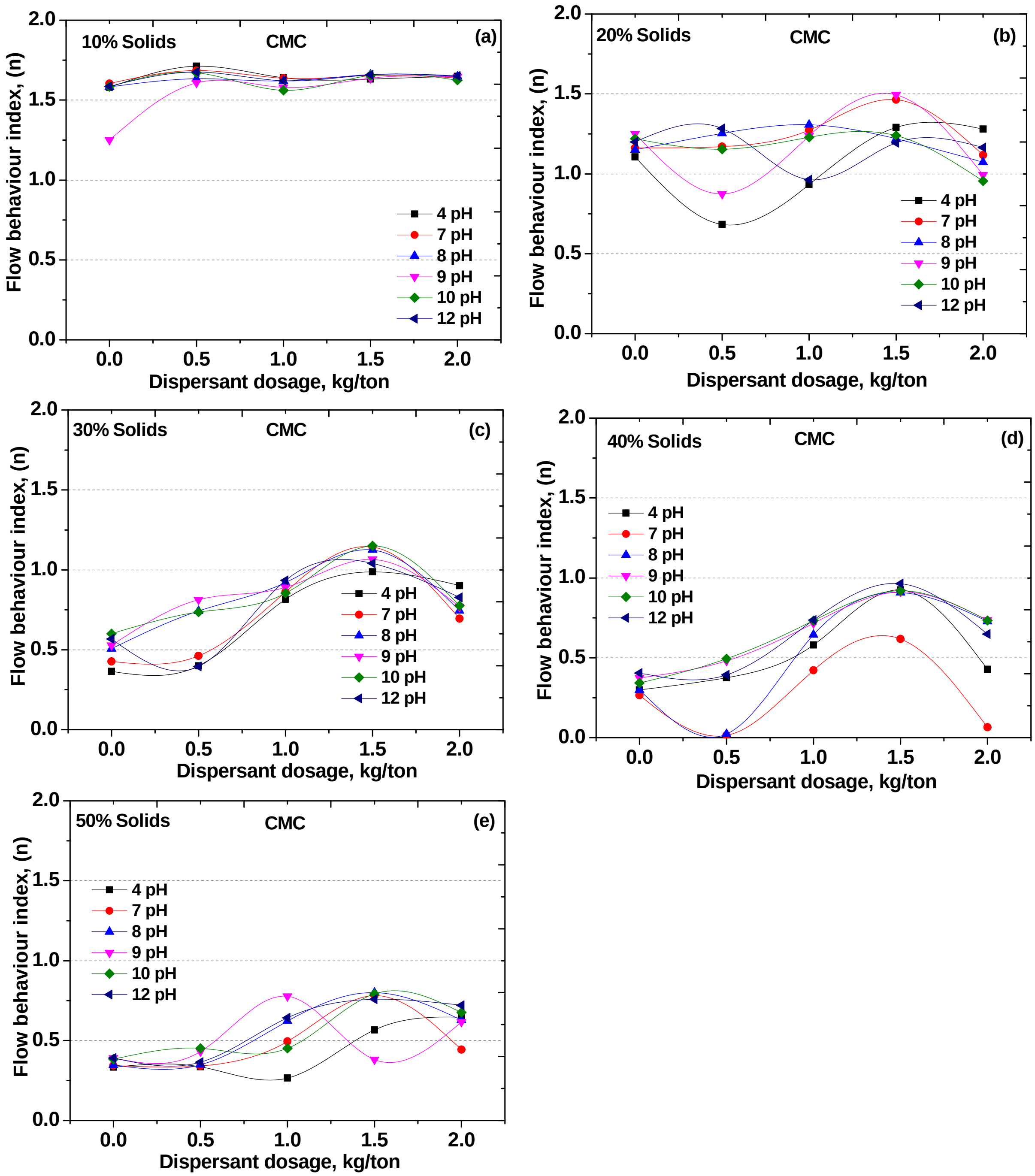


Figure 48. Effect of dispersant dosage (CMC) on flow behaviour index (n) for (a) 10, (b) 20, (c) 30, (d) 40 and (e) 50 percent solids at different pH.

4.2.6 Summary

The addition of polymeric anionic dispersant (CMC) has yielded to favourable pumpable characteristics. The role of carboxymethylcellulose (CMC) in favourable alteration of the rheological nature of the slurry for the coal variety indicates the suitability of dispersant for the coal in slurry transport application. The effect of dispersant up to 1.5 kg per ton dosage is clearly evident in reducing the shear stress values for 30 %, 40% and 50% solid loading for all pH values. The shear stress- shear rate relation did not alter with respect to dispersant dosage or pH at lower solid loadings (10 %, 20 %). At 10% solids concentration the exhibited shear thickening nature in the predictions based on power law model. The slurry with 20% and 30% solid loading showed transition from shear thickening to shear thinning behaviour with the increase in dispersant dosage and pH. For the higher solid concentration (40%,50%), the slurry exhibited shear thinning behaviour. For a given solids concentration, the flow behaviour index is increasing with increase in pH in many cases.

4.3 A comparative study on the rheological properties of two coal water slurries with Sodiumtripolyphosphate (STPP) as dispersant

4.3.1 Introduction

Rheology is an important research tool that can be used to characterise the ores/minerals based on the flow properties of the constituent gangue minerals. The flow characteristics of the coal water suspensions depends on physical and chemical properties of the coal. The maximum possible solids concentration for any CWS strongly depends on the coalification factors such as moisture, mineral matter, porosity hydrophobicity, organic matter content and oxygen containing functional groups.

This chapter describes the rheological behaviour of CWS with sodiumtripolyphosphate (STPP) as a dispersant was studied and compared for two coal water slurries prepared by *Coal 1* and *Coal 2*. The effectiveness of dispersant was investigated with respect to rheological characteristics and compared for two coal variety at shear rate in the range of 60-160 s^{-1} and at constant pH of 8. For all CWS of specific solids concentration (10%, 20%, 30%, 40% and 50%) and dispersant dosage (2.0, 4.0, 6.0 and 8.0 kg/ton) the shear stress values for shear rate in the range of 60-160 s^{-1} were obtained and compared at pH of 8. The variation of shear stress with shear rate and flow behaviour index (n) from power law model (refer equation (1) as shown in Chapter 2, Section 2.1.2.1) were used to compare the coal water slurries with respect to dispersant addition and solids loading.

4.3.2 Effect of solids concentration on *Coal 1* and *Coal 2* under STPP as dispersant

The rheological data plotted as the variation shear stress with respect to dispersant dosage (constant shear rate curves) are presented in Fig 49 (a, c, e, g, i) and Fig.49 (b, d, f, h, j) for *Coal 1* and *Coal 2* respectively. A Non-Newtonian behaviour was clearly evident at all coal concentrations.

For an increase in solids concentration, an increase in the shear stress values were seen. This behaviour can be well explained as, the increased molecular interaction with significant increase of friction among the coal particles [39]. The shear stress values are almost same at lower solids (10%,20%) concentration for all dispersant dosages tested for the two coals. However, at higher solids concentration (30%,40% and 50%), lower magnitude shear stress values for *Coal 1* are quite noticeable, for all dispersant loadings. The rheological properties of coal can be greatly influenced by the carbon content, ash, soluble ions, hydrophobicity, pore structure and distribution etc [9]. The high percentage of ash in the coal leads to the aggregation, which increases the viscosity of CWS [16]. The presence of soluble ions in coal like Ca^{2+}, Mg^{2+}, Al^{3+} and Fe^{3+} in the CWS favours the formation of cross-linking net structures by bridge bonding among the coals which can impart more stability to the slurry [57]. In addition, it can be noted that the *Coal 1* is relatively rich in carbon content than *Coal 2* (see Table 1).

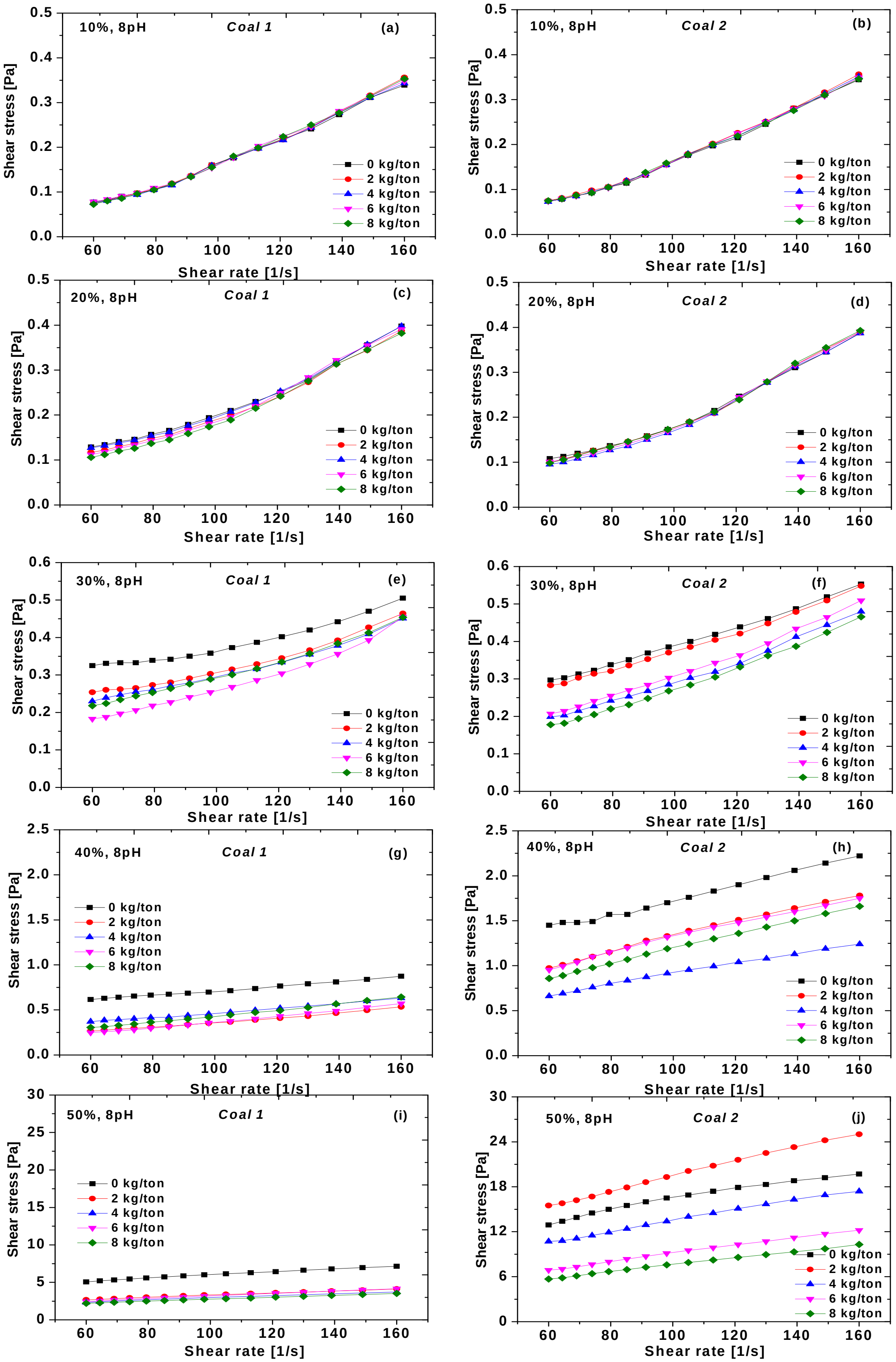


Figure 49. Shear stress versus shear rate on CWS with STPP as dispersant at different solids loading (a) 10%, (c) 20%, (e) 30%, (g) 40% and (i) 50% for *Coal 1,* and (b) 10%, (d) 20%, (f) 30%, (h) 40% and (j) 50% for *Coal 2.*

4.3.3 Effect of dispersant on *Coal 1* and *Coal 2* under STPP as dispersant

To understand the effect of dispersant dosage on shear stress values, the rheological data shown in Fig.49 (a, c, e, g, i) and Fig.49 (b, d, f, h, j) is cross plotted in Fig.50 (a, c, e, g, i) and Fig.50 (b, d, f, h, j) as the variation shear stress with respect to dispersant dosage (at constant shear rate of 60, 79.4, 98, 130 and 160 s^{-1}) for the 10%, 20%, 30%, 40% and 50% solids loading. For a given coal variety and dispersant dosage, at lower percentage of solids (10%, 20%), the shear stress versus shear rate relations are almost similar in nature with almost same magnitude of shear stress values with respect to a given dispersant dosage. The effect of dispersant is quite noticeable at the higher solids loading (30%, 40% and 50%).

For the CWS at 30% solids loading, for all shear rates tested, the shear stress attains minima at dispersant dosages of 6 kg/ton for *Coal 1* and 4 kg /ton for *Coal 2* respectively. In the case of 40% solids loading, for all shear rates tested the dispersant loadings, the shear stress attains minima at dispersant dosage of 4 kg/ton for both *Coal 1* and *Coal 2* respectively

For 50% CWS, the minimum shear stress values obtained for 2 kg/ton and upto 8 kg/ton of dispersant for *Coal 1* and *Coal 2* respectively. For a given solids concentration, an increase of shear stress with dispersant addition was observed after attaining the minima. The agglomeration of particles due to the excessive dispersant dosage can increase the ionic strength of the slurry, which results in formation of strong electrical double layers around the solid particles and thereby reduce the electrostatic repulsive forces among the particles. As a result, the shear stress increases with increase in dispersant loading after the saturation limit [55].

For a given coal variety and solids concentration, the magnitude of variation in shear stress with dispersant dosage was higher at lower values of shear rate, whereas for the highest shear rate tested (160 s^{-1}), the dispersant has no significant effect as the shear stress did not decrease much with the addition of dispersant. The decrease of shear stress at higher shear rate was due to the continuous breakdown of slurry structure and continuous or sudden breakdown of aggregates in the slurry [53].

4.3.4 Flow behaviour Index on *Coal 1* and *Coal 2* under STPP as dispersant

Fig.51 (a)-(e) shows the effect of dispersant dosages on flow behaviour index of different solids concentrations of CWS (10%, 20% ,30%, 40% and 50%) for *Coal 1* and *Coal 2* respectively. For a given coal variety, at 10% CWS, the flow behaviour index (*n)* values are very close in magnitude and for 20%-50% of CWS, change in the flow behaviour index (*n)* value with dispersant addition is noticeable. The CWS of 10% and 20% solids loading exhibited shear thickening behaviour (dilatant) for all dispersant dosages and CWS (*Coal 1* and *Coal 2*) tested. On the contrary, the CWS of 30%, 40% and 50% solids loading are shear thinning (pseudoplastic) in nature. The transition of flow behaviour from shear thickening to shear thinning was observed between 20-30% solids loading. CWS with better rank coal (*Coal 1*) was exhibited the low value of flow behaviour index (*n)* and shear thinning characteristics in comparison to *Coal 2.* Generally, shear thinning behaviour is favourable for the transportation of slurry owing to the decrease in the viscosity with the increase of shear rate.

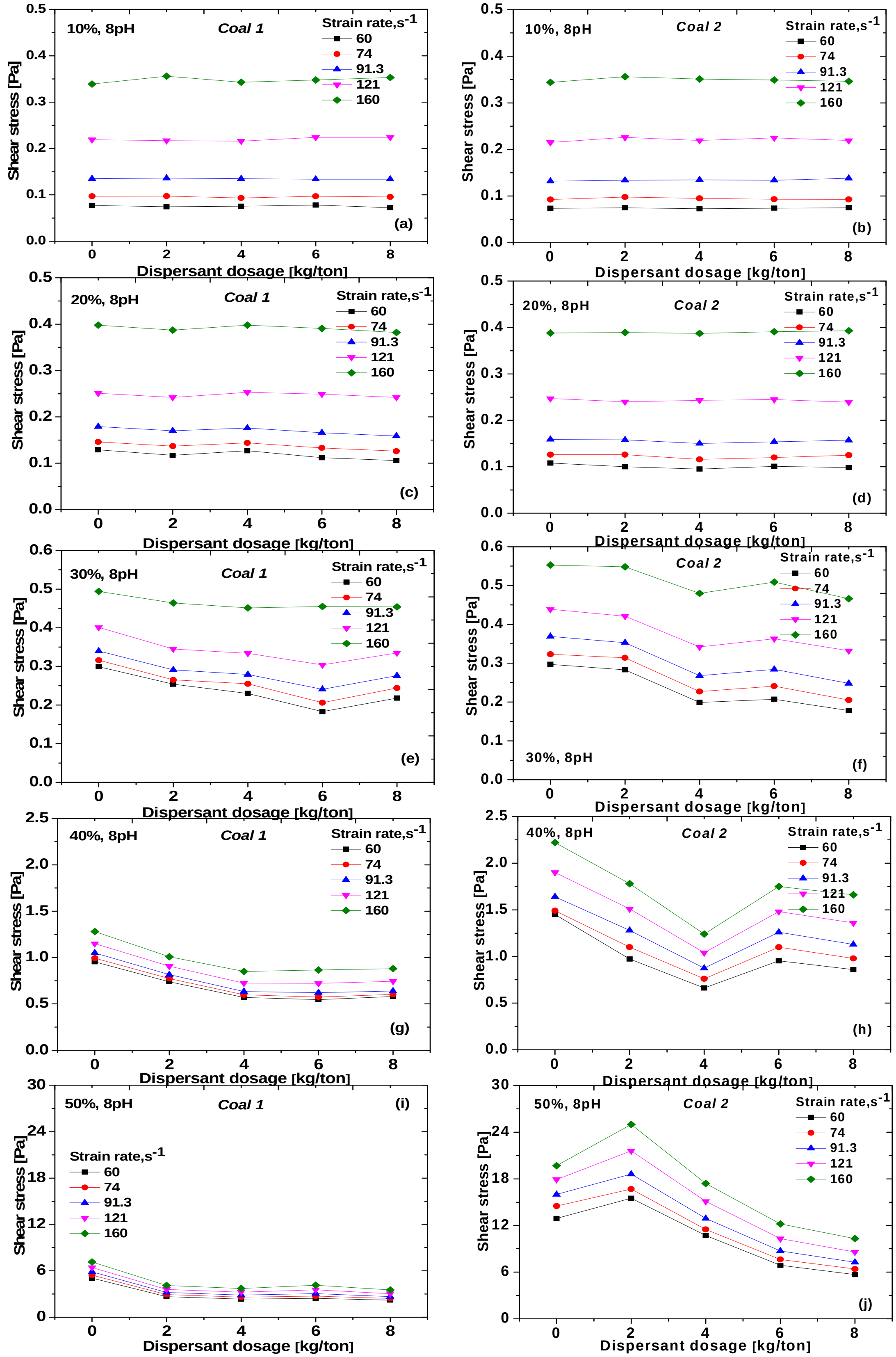


Figure 50. Effect of STPP dispersant dosage on CWS at different solids loading (a) 10%, (c) 20%, (e) 30%, (g) 40% and (i) 50% for *Coal 1,* and (b) 10%, (d) 20%, (f) 30%, (h) 40% and (j) 50% for *Coal 2*.

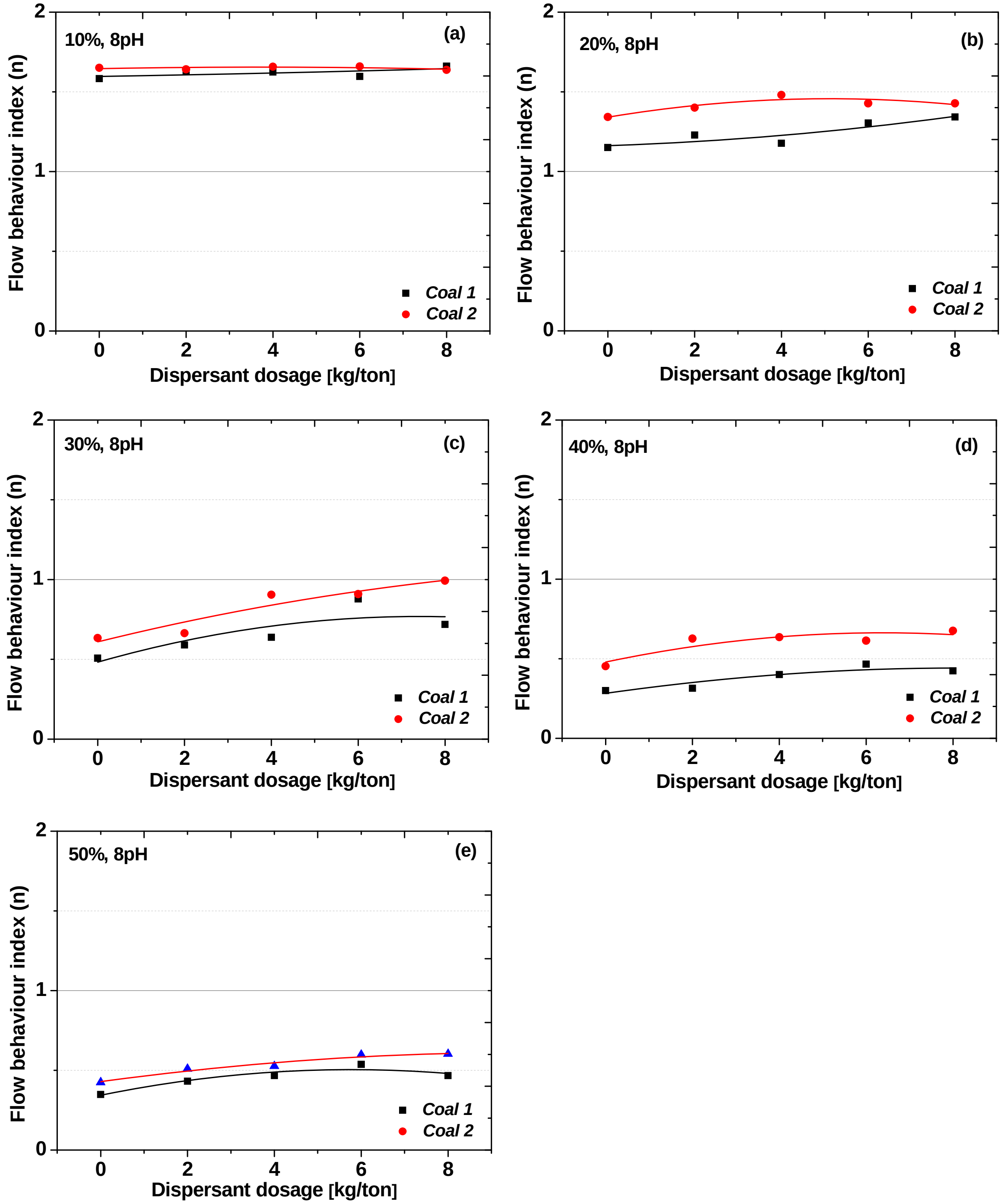


Figure 51. Effect of STPP on flow behaviour index of CWS (*Coal 1* and *Coal 2*) at different solids loading, (a) 10%, (b) 20%, (c) 30%, (d) 40% and (e) 50%.

4.3.5 Summary

While comparing the rheological behaviours of two different coals (*Coal 1* and *Coal 2*) with STPP as dispersant. The rheological data obtained in the shear rate range of 60-160 s^{-1} was successfully fitted for the power law model and flow behaviour index of each slurry was calculated. For lower solids loading (10% and 20%), the shear stress of the slurries did not change much with the gradual addition of dispersant and shear thickening nature was observed for these two solids loading. For the shear rates tested (60-160 s^{-1}), the transition from shear thickening to shear thinning nature was observed between 20% and 30% solids loading. In comparison, more favourable flow characteristics were observed for *Coal 1*. The reason for the same was the chemical composition and lower percentage of ash bearing matter in comparison to that of *Coal 2*.

4.4 A comparative study on the rheological properties of two coal water slurries with Carboxymethylcellulose (CMC) as dispersant

4.4.1 Introduction

A good understanding of the rheological properties of coal water slurries is essential for design and optimisation of the processes. The flow characteristics of the coal water suspensions depends on (1) physical and chemical properties of the coal such as ash content, the amount of inherent water, the degree of coal oxidation, and the quantity of surface-active functional groups. Using low rank coals having higher ash content, preparation of CWS of higher coal concentration with proper flowability is challenging due to the dominant presence of mineral matter.

In this chapter the rheological behaviour of CWS with carboxymethylcellulose (CMC) as a dispersant was studied and compared for two Indian coal water slurries prepared by using *Coal 1* and *Coal 2*. The effectiveness of dispersant was investigated with respect to rheological characteristics and compared for two coal variety. For all CWS of specific solids concentration (10%, 20%, 30%, 40% and 50%) and dispersant dosage (0.5, 1.0, 1.5 and 2.0 kg/ton) the shear stress values for shear rate in the range of 60-160 s^{-1} were obtained and compared at pH of 8. The variation of shear stress with shear rate and flow behaviour index (*n*) from power law model (refer equation (1) as shown in Chapter 2, Section 2.1.2.1) were used to compare the coal water slurries with respect to dispersant addition and solids loading.

4.4.2 Effect of solids concentration on *Coal 1* and *Coal 2* under CMC as dispersant

The variation of shear stress was measured for the coal water slurries of varying solid concentration (10%, 20%, 30%, 40% and 50%), at four distinct concentrations of the dispersant (0.5, 1.0, 1.5 and 2.0 kg/ton) for shear rates between 60-160 s^{-1} at constant pH of 8. The rheological data plotted as the variation shear stress with respect to dispersant dosage (constant shear rate curves) are presented in Fig.52 (a, c, e, g, i) and Fig.52 (b, d, f, h, j) for *Coal 1* and *Coal 2* respectively. A Non-Newtonian behaviour is clearly evident at all coal concentrations. For an increase in solids concentration, an increase in the shear stress values are seen. The behaviour can be well explained as the increased molecular interaction with significant increase of friction among the coal particles [39].

For the two coals, the shear stress values are almost same at lower solid concentrations for all dispersant dosages. However, at higher solids concentration, lower magnitude shear stress values for *Coal 1* are quite noticeable. The rheological properties of coal can greatly be influenced by the carbon content, ash, soluble ions, hydrophobicity, pore structure and distribution etc [9]. The degree of carbonisation increases the amount of carbonyl groups and leading to more hydrophobic nature of the surface. This effect results in favourable flow characteristics in the form of lower viscosity of the CWS [58]. The high percentage of ash in the coal leads to the aggregation which increases the viscosity of CWS [16]. The presence of soluble ions in coal like Ca^{2+}, Mg^{2+}, Al^{3+} and Fe^{3+} in the CWS favours the formation of cross-linking net structures by bridge bonding among the coals which can impart more stability to the slurry [57]. Hence the higher shear stress values for *Coal 2* in comparison to *Coal 1* is possibly due to the presence of ash bearing minerals in the former. In addition, *Coal 1* is relatively richer in carbon content than *Coal 2* (see Table 1).

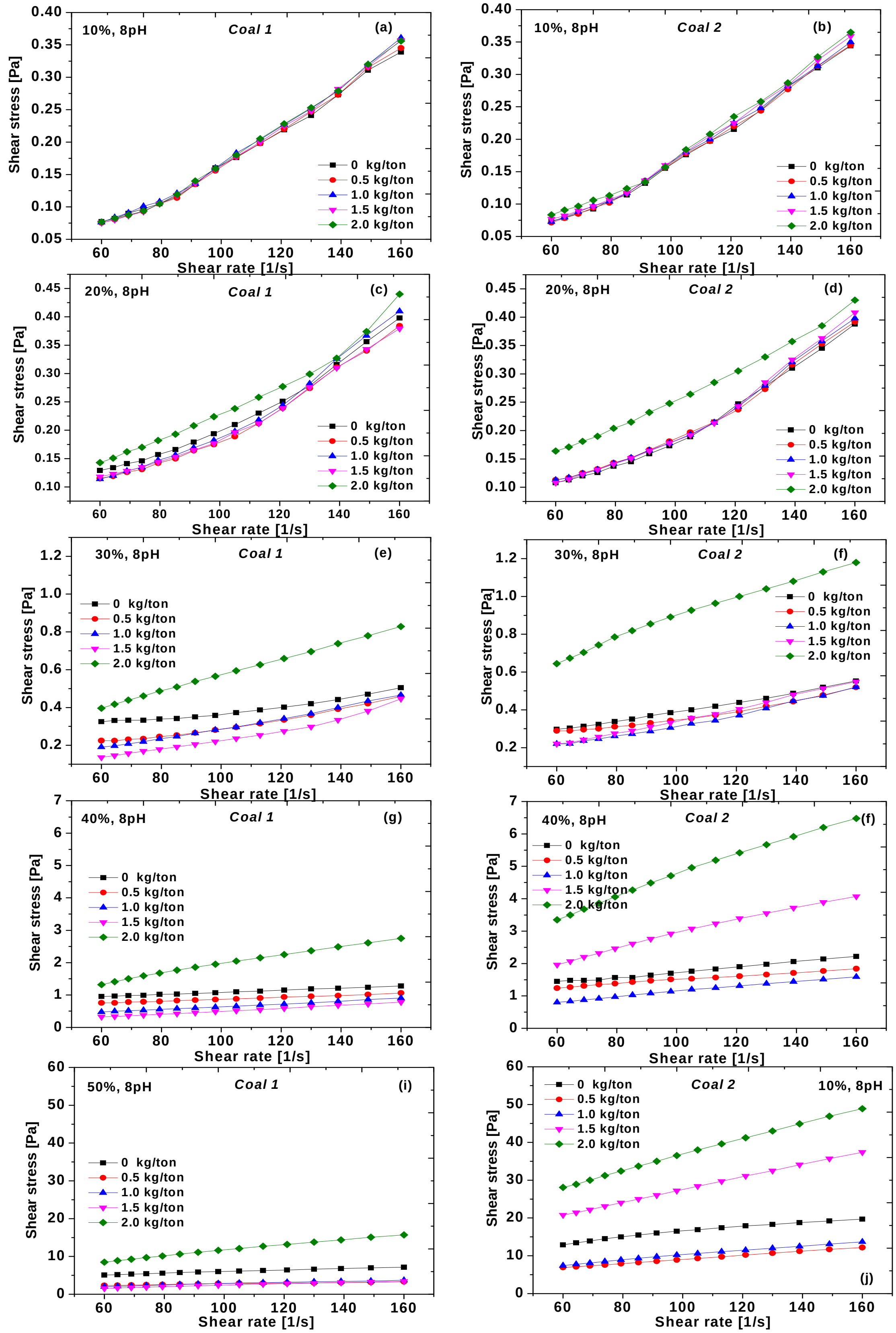


Figure 52. Shear stress versus shear rate for CWS with CMC as dispersant at different solids loading (a) 10%, (c) 20%, (e) 30%, (g) 40% and (i) 50% for *Coal 1,* and (b) 10%, (d) 20%, (f) 30%, (h) 40% and (j) 50% for *Coal 2*.

4.4.3 Effect of dispersant on *Coal 1* and *Coal 2* under CMC as dispersant

To understand the effect of dispersant dosage on shear stress values, the rheological data shown in Fig.52 (a, c, e, g, i) and Fig.52 (b, d, f, h, j) is cross plotted in Fig.53 (a, c, e, g, i) and Fig.53 (b, d, f, h, j) as the variation shear stress with respect to dispersant dosage (at constant shear rate of 60, 74, 91.3, 121 and 160 s^{-1}) for the 10%, 20%, 30%, 40% and 50% solid loadings for *Coal 1* and *Coal 2* respectively. For both types of coal, at lower percentage of solids (10%, 20%), the shear stress versus shear rate relations are similar in nature with almost same magnitude of shear stress values with respect to a given dispersant dosage. The effect of dispersant is quite visible at the higher solid loadings (30%, 40% and 50%).

For the CWS (*Coal 1* and *Coal 2*) at 30% solid loading, for all shear rates tested, the shear stress values attain minima at dispersant dosages of 1.5 and 1.0 kg /ton respectively. For the highest shear rate tested (160 s^{-1}), the dispersant has no significant effect as the shear stress values do not decrease much with the addition of dispersant. In the case of 40% solid loading, for all shear rates tested the dispersant loadings, the shear stress attains minima at dispersant dosage of 1.5 and 1.0 kg/ton dispersant addition respectively for *Coal 1* and *Coal 2*.

For 50% CWS, at a shear rate of 60, 74, 91.3, 121 s^{-1}, the minimum shear stress values obtained for 1.5 kg/ton and 0.5 kg/ton of dispersant for *Coal 1* and *Coal 2* respectively. For the CWS (*Coal 1*), at the highest shear rate tested (160 s^{-1}), the minimum shear stress values are obtained at 0.5 kg/ton dosage loading and further dispersant addition has no significant effect on the magnitude of shear stress for 50% solid loading. In the case of CWS (*Coal 2*), minimum shear rate is obtained at a dispersant dosage of 0.5 kg/ton at shear rate of 160 s^{-1}.

The surface chemistry of the suspension particle plays major role in attributing the rheological properties of the slurries. The nature and amount of dispersant added to the slurry and subsequent generation of surface charge on the suspended particle greatly influences the rheological properties. For a given percent solids and coal, an increase of shear stress versus shear rate was observed after attaining the minimum value of the shear stress. This can be explained as, the agglomeration of particles due to the excessive dispersant dosage can increase the ionic strength of the slurry, which results in formation of strong electrical double layers around the solid particles and thereby reduce the electrostatic repulsive forces among the particles. As a result, the shear stress values increase with increase of dispersant loading after the saturation limit [55-56]. For the higher solid loadings of 40% & 50%, addition of dispersant beyond 1.0 kg/ton has increased the shear rate values for *Coal 2*. For *Coal 1*, the same phenomena is seen after 1.5 kg/ton of dispersant. It indicates that *Coal 1* is susceptible to the adsorption of CMC on the surface as it contains more carbon content in comparison to *Coal 2*. For the two coals tested, it can be noted that for a given percent solids and dispersant dosage, a decrease in shear stress is seen at higher shear rate values. This is due to the continuous breakdown of slurry structure or continuous and sudden breakdown of aggregates in the slurry [39].

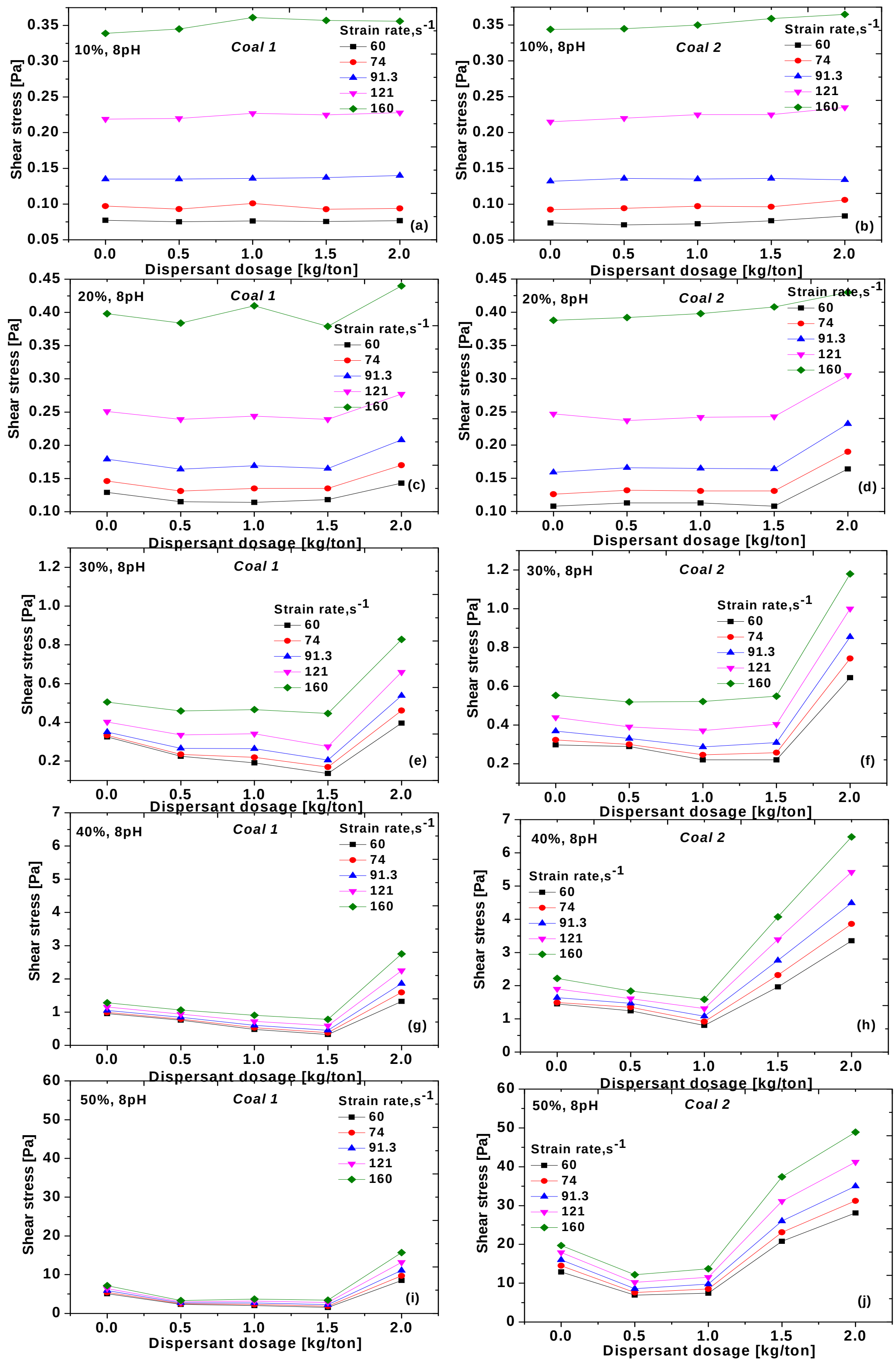


Figure 53. Effect of CMC on dispersant dosage of CWS at different solids loading (a) 10%, (c) 20%, (e) 30%, (g) 40% and (i) 50% for *Coal 1,* and (b) 10%, (d) 20%, (f) 30%, (h) 40% and (j) 50% for *Coal 2.*

4.4.4 Flow behaviour Index on *Coal 1* and *Coal 2* under CMC as dispersant

Fig.54 (a-e) shows the flow behaviour index values are plotted for all CWS with respect to solids concentration for *Coal 1* and *Coal 2*. For 10% CWS, the flow behaviour index values are very close in magnitude and the slurries are dilatant in nature. For 20%-50%, the change in the flow behaviour index value with dispersant addition is noticeable. Interestingly, CWS of 10% and 20% solid loadings exhibited shear thickening behaviour for all dispersant dosages and CWS (*Coal 1* and *Coal 2*) tested. On the contrary, the CWS of 30%, 40% and 50% solid loading are shear thinning in nature. The transition of flow behaviour from shear thickening to shear thinning was observed between 20-30% solids loading. Generally, shear thinning behaviour is favourable for the transportation of slurry owing to the decrease in the viscosity with the increase of shear rate. For a solids loading and dispersant dosage, the wide range of distribution of flow behaviour index is observed for *Coal 1* in comparison to *Coal 2*.

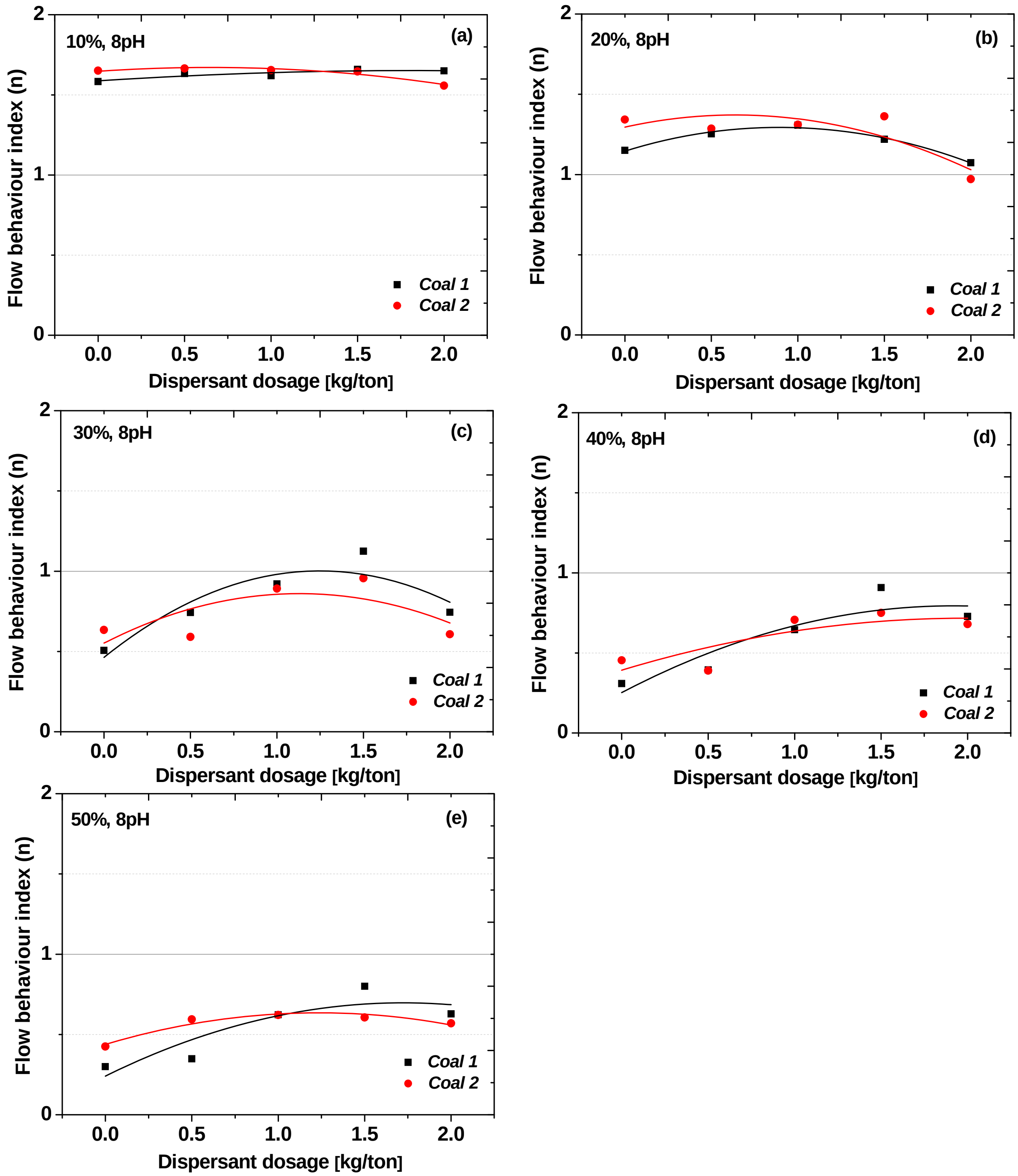


Figure 54. Effect of CMC on flow behaviour index of CWS (*Coal 1* and *Coal 2*) at different solids loading, (a) 10%, (b) 20%, (c) 30%, (d) 40% and (e) 50%.

4.4.5 Summary

The rheological behaviour of CWS is investigated and compared for two different Indian coals (*Coal 1* and *Coal 2*) with respect to solids loading, dispersant addition, at constant pH-8 in the shear rate range of 60-160 s^{-1}. For a given solid loading, addition of dispersant beyond 1.5 kg/ton and 1.0 kg/ton has increased the shear rate values for *Coal 1* and *Coal 2* respectively. The shear stress- shear rate relation did not alter with respect to dispersant dosage at lower solid loadings (10 %, 20 %) and they exhibited shear thickening nature as predicted by the fitting of the shear stress-shear rate data with power law model. The slurry with 30% solid loading showed transition from shear thickening to shear thinning nature with the increase in dispersant dosage. For the higher solid loadings, the slurry exhibited shear thinning behaviour with the addition of dispersant. A lower magnitude of shear stress versus shear rate, a wider distribution of flow behaviour index and favourable slurry pumpable characteristics were seen for *Coal 1* in comparison to *Coal 2* owing to the differences in the presence of ash or mineral bearing content.

4.5 Rheological Behaviour of coal water slurries of Indian coal using Carboxymethylcellulose and Sodiumtripolyphosphate as dispersant – A comparative study

4.5.1 Introduction

A higher coal concentration, minimum viscosity, lower yield stress and minimum settling were an essential requirement in preparation of CWS to facilitate the ease of handling during preparation, storage and transportation. Chemical additives are important ingredients and are added to reduce the viscosity, to maintain the fluidity and to improve the stability of CWS by inducing electrostatic or steric repulsions resulted by more negative charge on the coal surface [59]. An anionic dispersant can comparatively generate more negative charge on the coal surface by (1) flat adsorption of the hydrophobic tail portion of reagent over the coal surface with negatively charged head protruding out from the surface, (2) formation of stable surface complex with coal inorganic high valance cations (e.g. Al^{3+}, Fe^{3+}). Anionic polymeric dispersants in particular have been found to be more effective additives in stabilising the coal water slurries by introducing more surface charge by combination of steric and electrostatic repulsive forces. Much of the investigations suggests the use of sodiumtripolyphosphate (STPP) as an anionic dispersant and carboxymethylcellulose (CMC) as an anionic polymeric dispersant, to reduce the viscosity of coal water slurries and to improve the flow properties

In the present chapter discussed the study of rheological characteristics of CWS with addition two dispersants namely carboxymethylcellulose (CMC) and sodiumtripolyphosphate (STPP) for Indian coal variety (*Coal 1*). The effectiveness of dispersant addition is investigated and compared with respect to flow characteristics. The variation of shear stress with shear rate and flow behaviour index (n) from the power law model (refer equation (1) as shown in Chapter 2, Section 2.1.2.1) were used to compare the coal water slurries with respect to type of dispersant, dispersant dosage and solids loading at constant pH of 8.

4.5.2 Effect of solids concentration on *Coal 1* under STPP and CMC as dispersant

The variation of shear stress was measured for CWS at varying solids concentration (10%, 20%, 30%, 40% and 50%), at four distinct concentrations of the dispersant (0.5, 1.0, 1.5 and 2.0 kg/ton) and (2, 4, 6 and 8 kg/ton) for dispersants namely CMC and STPP respectively. All the tests were conducted at constant shear rates between 60-160 s^{-1} at constant pH of 8. The rheological behaviour was measured and compared for CMC and STPP.

The effect of shear stress on shear rate for CWS with addition of CMC and STPP as dispersants are shown in Fig.55 (a, c, e, g, i) and Fig.55 (b, d, f, h, j) respectively. A Non-Newtonian behaviour was clearly evident at all dispersant dosages and coal concentrations. An increase in the shear stress with an increase in solids concentration was seen. The increased molecular interaction results the significant increase of friction among the coal particles can be prime reason for the same [38].

For a given dispersant, the shear stress values are almost same at lower solids concentration for all dispersant dosages tested. However, at higher solids concentration, lower magnitude shear stress values for CWS with CMC as a dispersant are quite noticeable in comparison to STPP as a dispersant. The rheological properties of CWS with a type of dispersant can greatly influenced by nature of surface charge of the coal particle by the addition of dispersant. Anionic polymer-based dispersants are much more effective in reducing the viscosity of the CWS due to their capability to introduce coal surface charge with more negative values by a combination of steric effects and electrostatic repulsion [19]. In addition, it can be also noted that the CMC is an anionic polymeric dispersant and found relatively effective in reducing the viscosity of CWS in comparison to STPP.

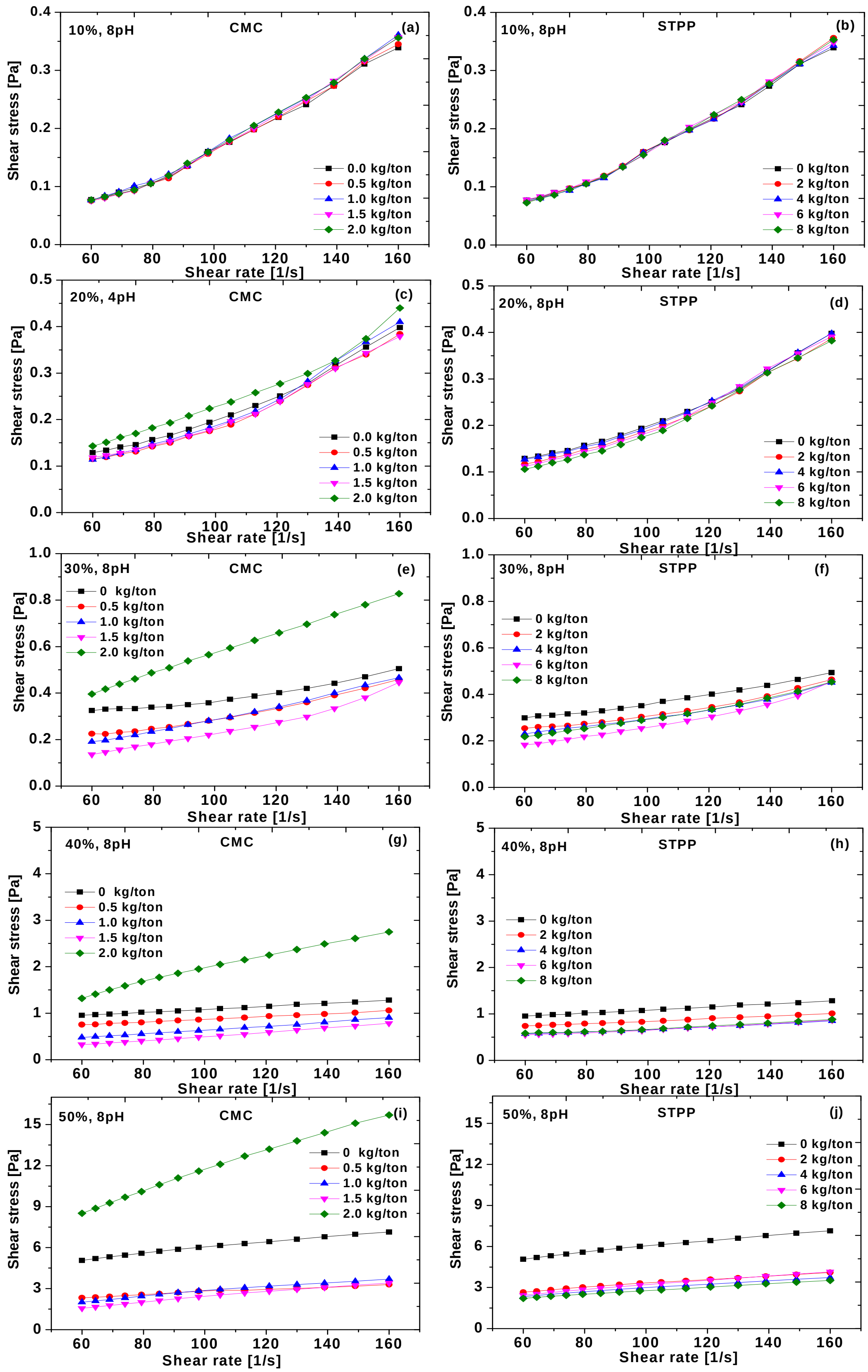


Figure 55. Shear stress versus shear rate on CWS at different solids loading (a) 10%, (c) 20%, (e) 30%, (g) 40% and (i) 50% for CMC, and (b) 10%, (d) 20%, (f) 30%, (h) 40% and (j) 50% for STPP.

4.5.3 Effect of dispersant on *Coal 1* under STPP and CMC as dispersant

To understand the effect of dispersant dosage on shear stress, the rheological data shown in Fig.55 (a, c, e, g, i) and Fig.55 (b, d, f, h, j) was cross plotted in Fig.56 (a, c, e, g, i) and Fig.56 (b, d, f, h, j) as the variation of shear stress with respect to dispersant dosage (at constant shear rate of (60, 74, 91.3, 121 and 160 s^{-1}) for 10%, 20%, 30%, 40% and 50% solids loading. For a given dispersant and its dosage, at lower percentage of solids (10%, 20%), the shear stress versus shear rate relations are almost similar in nature with respect to magnitude of shear stress. The effect of dispersant is quite noticeable at the higher solids loading (30%, 40% and 50%).

For the CWS at 30% solids loading, for all shear rates tested, the shear stress attains minima at dispersant dosages of 1.5 kg/ton for CMC as a dispersant and 6 kg /ton for STPP as a dispersant respectively. In the case of 40% solids loading, for all shear rates tested the dispersant loadings, the shear stress attains minima at dispersant dosage of 1.5 kg/ton and between 4 kg/ton to 6 kg/ton for CMC and STPP as a dispersant respectively.

For 50% CWS, the minimum shear stress values obtained for 1.5 kg/ton and between 2 kg/ton to 8 kg/ton of dispersant for CMC and STPP respectively. For a given dispersant and solids concentration, an increase of shear stress with dispersant addition was observed after attaining the minima. The agglomeration of particles due to the excessive dispersant dosage can increase the ionic strength of the slurry, which can result in formation of strong electrical double layers around the solid particles and thereby reduce the electrostatic repulsive forces among the particles. As a result, the shear stress increases with increase in dispersant dosage after the saturation limit [55].

For a given dispersant and at 30% solids concentration, the magnitude of variation in shear stress with dispersant dosage was higher at lower values of shear rate, whereas for the highest shear rate tested (160 s^{-1}), the dispersant has no significant effect as the shear stress did not decrease much with the addition of dispersant. The decrease of shear stress at higher shear rate was due to the continuous breakdown of slurry structure and continuous or sudden breakdown of aggregates in the slurry [39].

4.5.4 Flow behaviour Index on *Coal 1* under STPP and CMC as dispersant

Fig.57 (a-e) shows the effect of dispersant dosages on flow behaviour index of different solids concentrations of CWS (10%, 20%, 30%, 40% and 50%) for CMC and STPP respectively. For a given dispersant, at 10% CWS, the flow behaviour index (*n)* values are very close in magnitude and for 20%-50% of CWS, change in the flow behaviour index (*n)* value with dispersant addition is noticeable. For a given dispersant, the CWS of 10% and 20% solids concentration exhibited shear thickening behaviour (dilatant) for all dispersant dosages tested. On the contrary, the CWS of 30%, 40% and 50% solids concentration are shear thinning (pseudoplastic) in nature. The transition of flow behaviour from shear thickening to shear thinning was observed between 20-30% of solids concentration. For a given solids concentration, CWS with CMC as a dispersant was exhibited a higher and wider distribution flow behaviour index (*n)* in comparison to STPP. Generally, shear thinning behaviour is favourable for the transportation of slurry owing to the decrease in shear stress with the increase of shear rate.

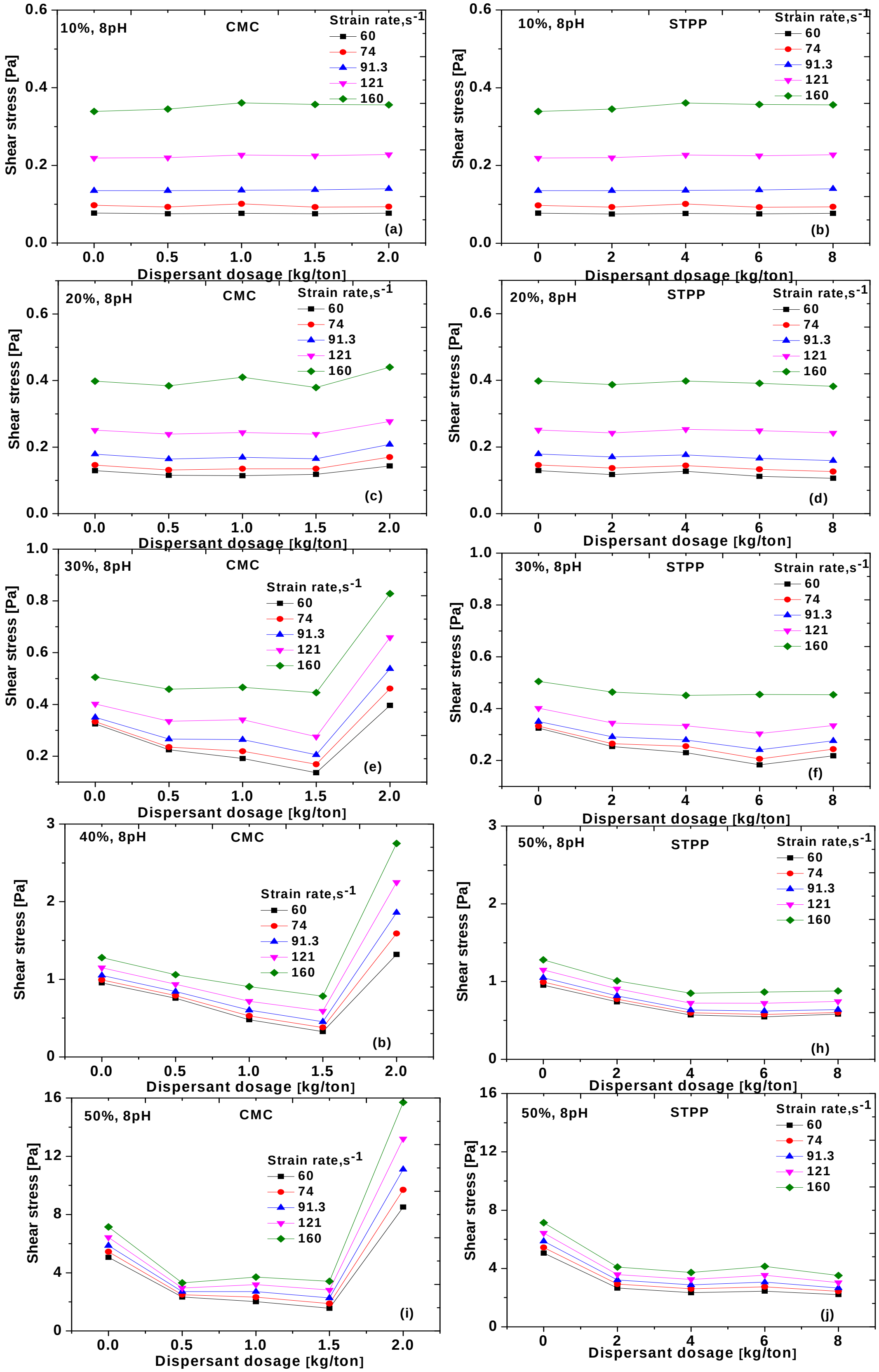


Figure 56. Effect of dispersant dosage on CWS at different solids loading (a) 10%, (c) 20%, (e) 30%, (g) 40% and (i) 50% for CMC, and (b) 10%, (d) 20%, (f) 30%, (h) 40% and (j) 50% for STPP.

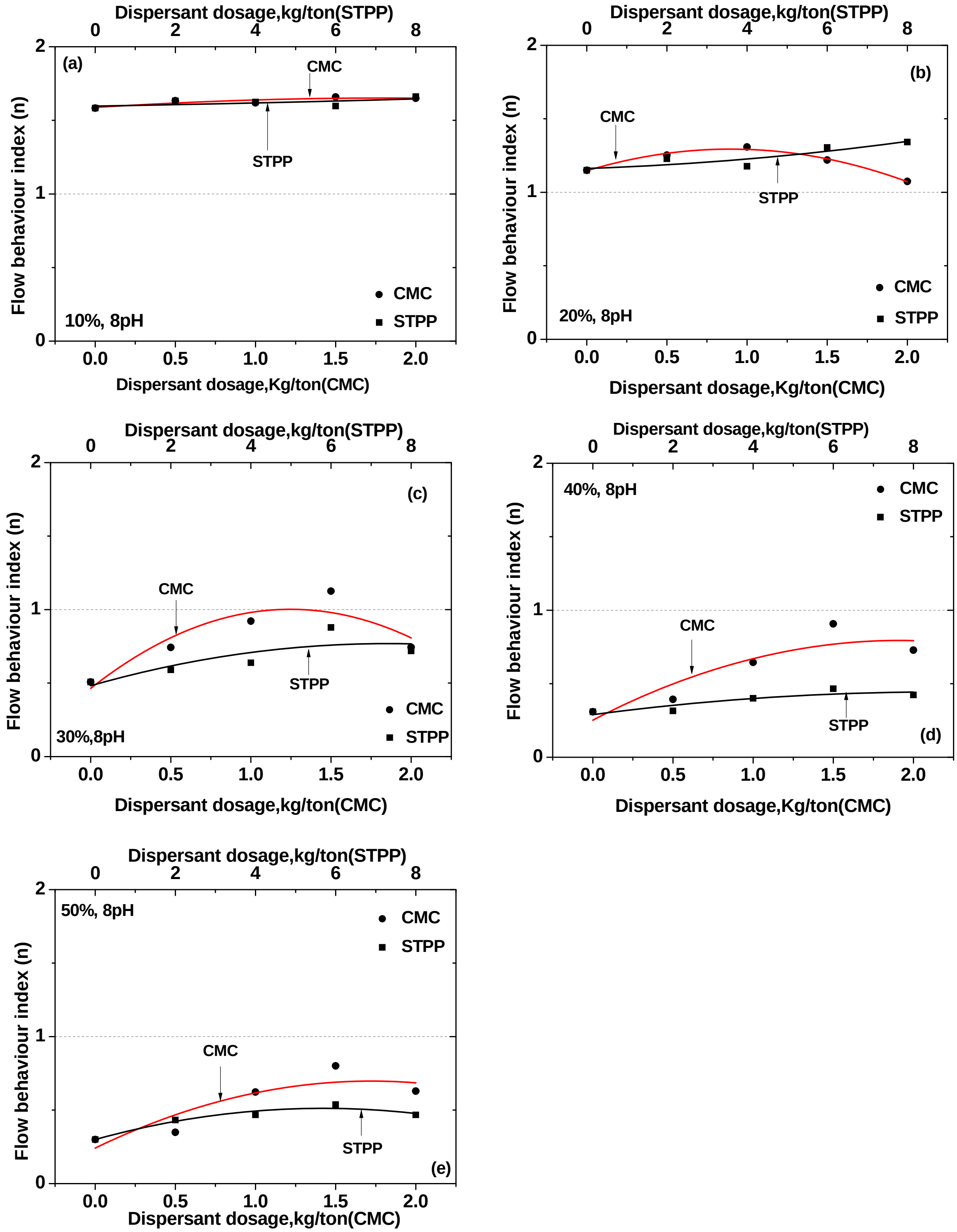


Figure 57. Effect of flow behaviour index on CWS at different solids loading, (a) 10%, (b) 20%, (c) 30%, (d) 40% and (e) 50% for CMC and STPP.

4.5.5 Summary

The rheological behaviour of the CWS was investigated for two different dispersants namely CMC (polymeric) and STPP (non-polymeric) and compared the effectiveness of dispersant addition. The rheological data was obtained in the shear rate range of 60-160 s^{-1} was successfully fitted for the power law model and flow behaviour index of each slurry was calculated. For lower solids concentration (10% and 20%), the shear stress of the slurries did not change much with the addition of dispersant and shear thickening nature was observed. For the shear rates tested (60-160 s^{-1}), the transition from shear thickening to shear thinning nature was observed between 20% and 30% solids loading. For a given solids concentration, the lower values of shear stress were reported for CMC as a dispersant in comparison to STPP. For a given solids concentration, the dispersant dosage required to attain per unit shear stress was lower for CMC in comparison to STPP. The coal surface with more negative surface charge resulted by the combination of steric effects and electrostatic repulsion was prime reason for the same.

CHAPTER-5

CHAPTER-5

CONCLUSIONS AND SCOPE OF FUTURE WORK

5.1 Conclusions

Based on the present investigation carried out, the following conclusions are drawn.

1) Proximate and ultimate analysis conform the lower ash content in *Coal 1* in comparison to *Coal 2*. The SEM and XRD analysis revealed the presence of the particles containing both carbonaceous and non-carbonaceous mineral matter.

2. The zetapotential and turbidity measurements indicated the suitability of CMC and STPP as dispersant for the CWS prepared using the two coal varieties. The dispersant addition is much effective for *Coal 1* in comparison to *Coal 2*. Zetapotential and turbidity conforms that the CMC is much effective dispersant in comparison to STPP for the coal variety tested.

3. Sodium tripolyphosphate (STTP) and Carboxymethylcellulose (CMC) favourably altered the rheological nature of the slurry for the coal variety indicating their suitability as dispersants for the coals in slurry transport application.

4. The shear stress- shear rate relations did not alter with respect to dispersant dosage or pH at lower solid loadings (10%, 20%) and they exhibited shear thickening nature in the predictions based on power law model. Interestingly, the slurry with 30% solid loading showed transition from shear thickening to shear thinning behaviour with the increase in dispersant dosage and pH. For the higher solid loadings, the slurry exhibited shear thinning or pseudoplastic behaviour at higher pH values with the addition of dispersant.

5. For a given dispersant and percentage of solids, a lower magnitude of shear stress versus shear rate and favourable slurry pumpable characteristics were seen for *Coal 1* in comparison to *Coal 2* owing to their chemical nature and amount of ash bearing mineral constituents present.

6. For a given solids concentration, the dispersant dosage required to attain per unit shear stress was lower for CMC (anionic polymeric dispersant) in comparison to STPP (anionic dispersant). The coal surface with more negative surface charge resulted by the combination of steric effects and electrostatic repulsion was prime reason for the same.

5.2 Scope of Future Work

In countries like India where high-quality coal is scarce, the successful pre-processing and economic transportation of the coal for further and efficient utilization should be seen as an important step. The extracted coal needs to be stored, handled and transported for any subsequent mineral processing unit operations. The transportation of coal as CWS is an effective transportation method as it can be carried out through pipes with minimum cost and energy consumption. Using low rank coals having higher ash content, preparation of CWS of higher coal concentration with proper flowability is a challenging task due to the dominant presence of mineral matter.

The major requirement in preparation of CWS is that it should have higher coal concentration with minimum viscosity to allow ease of handling during preparation, storage and transportation. Chemical additives are important ingredients in reducing the viscosity, maintaining fluidity and improving the stability of CWS by introducing the electrostatic or steric repulsions or increasing the steric wettability of coal. The extant literature over the subject reveals that the identification or synthesis of suitable chemical dispersant is an influential factor for achieving favourable rheological characteristics.

Based on the outcome of the present investigation, process methodologies can be developed in industrial scale slurry transportation. The study of the rheological behaviour of an Indian low rank coals is still a thrust area of research. The rheological behaviour of CWS at different strain rates can be studied and database can be established. The research methodologies can be explored to use of CWS with higher solids concentration for transportation application. The synthesis and use of eco-friendly and natural dispersants in preparation of CWS can be focussed for environmentally friendly storage and transportation system.

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LIST OF PUBLICATIONS

INTERNATIONAL JOURNALS

1. M. Ananda Rao, M. V. Pavan Kumar, S. Subba Rao & N. Narasaiah (2018) Rheological behaviour of coal-water slurry using sodium tripolyphosphate as a dispersant, International Journal of Coal Preparation and Utilization, DOI: 10.1080/19392699.2018.1485664

2. M. Ananda Rao, M. V. Pavan Kumar, S. Subba Rao & N. Narasaiah (2018) Rheological behaviour of coal-water slurries of Indian coals using carboxymethylcellulose as dispersant- a comparative study, International Journal of Coal Preparation and Utilization, DOI:10.1080/19392699.2018.1518901

3. M. Ananda Rao, Veerapuram Yerriswamy, S. Subba Rao & N. Narasaiah.A Comparative Study on the Rheological Properties of Two Coal Water Slurries with Sodiumtripolyphosphate as Dispersant "International Journal of Coal Preparation and Utilization" (Under review)

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