EFFECT OF MIXTURE OF A NON-IONIC AND A CATIONIC SURFACTANT FOR PREPARATION OF STABILIZED HIGH CONCENTRATION COAL WATER SLURRY

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Introduction

Consequent on depletion of oil reserve, alternate source of energy production from biodiesel (Chen and Chen 2011) and biomass (Manganaro et al. 2011) has been taken up. But the availability and efficiency of all these resources in comparison to coal are not economical and aplenty. Hence industries opt for coal as cheap and easy source of energy production and extensive research has been taken up in the field of coal water slurry (CWS) (Mishra and Kanungo 2000).

Low viscosity and high loading of coal particles are the main guiding factors for efficiencyof CWS (Das et al. 2008). High concentrated CWS involves inter-particle interaction whichleads to high viscosity and creates problems during pipeline transportation (Lee 2007). In order to reduce the interaction between the particles, different additives have been tried worldwide to enhance the rheological behavior of CWS (Li et al. 2017; Shen et al. 2008; Gurses et al. 2006). Some researchers have carried out studies on concentrated suspensions with dosages ofnatural additives to achieve slurry stabilization as well as viscosity reduction (Senapati et al.2008; Das et al. 2009; Routray et al. 2018). Others have tried only with chemical additives or both for behavioral study of the slurry (Das et al. 2010; Ongisirimongkol and Narasingha 2012; Yi, Gopan, and Axelbaum 2014; Zhu et al. 2014, 2012). Mixture of additives has an important role in the stabilization of high concentrated CWS. Due to the synergetic interaction between the two additives, the performance is improved and such interdependent behavior of mixture of additives reduces the amount of additive required for the CWS stabilization. Das et al. (2013) has prepared a stable coal water dispersion using a mixture of saponin isolated from *Sapindous lauifolia* and CTAB or SDS and the results indicated that the mixture of additives was found to be more effective than single additive system. It has also been indicated in literature that saponin isolated from *Acacia concina* commonly known as shikakai is more surface active than saponin isolated from *Sapindous lauifolia* (Das et al. 2009).

The rheological behavior of coal water mixture largely depends upon the concentration of coal (Dmitrienko, Nyashina, and Strizhak 2018), as well as particle size distribution (Saddler et al. 1991). Studies indicated that the particle size and size distribution plays a major role in the rheological behavior of the coal ash slurry (Pani et al. 2015; Senapati, Mishra, and Parida 2010; Senapati et al. 2011, 2012). In monomodal distribution, there is less chance for achieving high concentration as it leaves scope for space between the particles. The particle size should be such that the space between the coal particles should be reduced. So bimodal concept is a necessity where the coal particles of two distinct fractions are used so as to enhance the coal concentration. As the packing efficiency increases at constant solid loading, less water is required to fill the inter particle voids. This makes a larger fraction of water available to increase separation distance between the particles thereby reducing the viscosity. So far very little work has been done for preparation of high concentrated CWS taking bimodal distribution of Indian coal with applications of mixture of chemical and bio-additives. This finding is not only important for reduction of viscosity of coal slurry, but also for reducing viscosity in other samples like drug delivery (Rutkowski et al. 2018; Mu et al. 2019; Rutkowski et al. 2019a, 2019b).

In our previous paper, we have studied the synergestic interaction between the non-ionic surfactant saponin isolated from shikakai and a anionic surfactant sodium do-decyl benzene sulfonate (Routray et al. 2019). The objective of this paper is to study the interaction of saponin of shikakai with a cationic surfactant, do-decyl ammonium bromide, and their combined effect in the stabilization of a high concentration CWS containing four types of bimodal coal sample.

Experimental Section

Size of Coal Sample

The coal samples used in this study have been collected from Nandira mines, India. The samples were ground in a laboratory ball mill with different grinding time to prepare fine and coarse fraction sizes. Fine fractions of sizes below 38 µm were mixed separately with four distinct coarse fractions of 212–300 µm, 90–300 µm, 150–212 µm, and 75–150 µm and these four representative bimodal samples were labeled as S-1, S-2, S-3, and S-4, respectively. Also, the weight ratio of coarse to fines in sample S-1 was varied in different proportions like 0.7:0.3, 0.65:0.35, and 0.6:0.4 for rheological measurements. Tables 1 and 2 show the proximate and ultimate analysis of coal samples, respectively. Malvern Particle size analyzer was used to measure the particle size distribution of the coal samples. The particle size

Table 1. Proximate analysis of coal sample.

Moisture (%)	5.76
Ash (%)	37.89
Volatile matter (%)	24.22
Fixed carbon (%)	32.13

Table 2. Ultimate analysis of coal sample.

Carbon (%)	84.26
Hydrogen (%)	4.78
Nitrogen (%)	1.75
Oxygen (%)	8.66
Sulfur (%)	0.55

distribution of four bimodal samples S-1, S-2, S-3, and S-4 are given in Figure 1 and d_{10}, d_{50}, d_{90} data of the particle size distribution of all samples are given in Table 3.

Sources of Additives

Acacia Concinna fruits have been collected from Koraput and Paralakhemundi areas situated in eastern part of India where these plants are grown plentily. The saponin extracted from dried Acacia concinna by the solvent extraction method is commonly known as Shikakai (Das et al. 2009). The structure of Shikakai with a molecular mass of 1,058 is shown in Figure 2 (Das et al. 2009). Di-docyl ammonium bromide (DDAB) is a commercial chemical purchased from Sigma-Aldrich, and was used for the study without any further purification. The structure of DDAB is given in Figure 3.

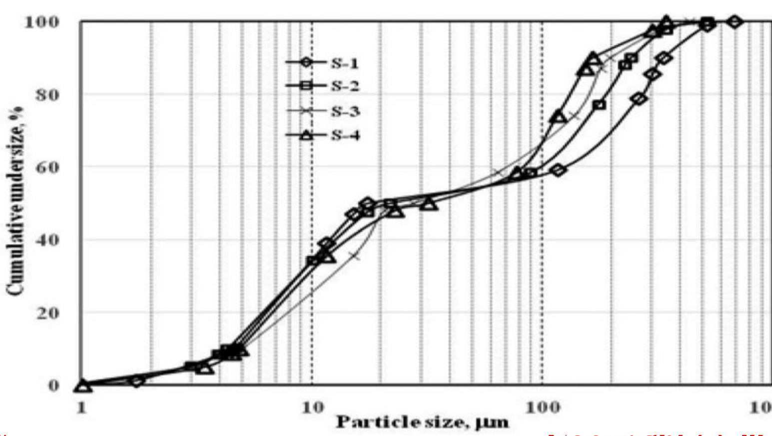

Figure 1. Particle size distribution of all four coal samples S-1, S-2, S-3, and S-4.

Table 3. Particle size distribution of coal samples (S-1, S-2, S-3, and S-4).

Sample	d_{10}	d_{50}	d_{90}
S-1	4.6	17.46	333.6
S-2	4.3	21.7	242.4
S-3	4.96	27.6	196.5
S-4	4.8	31.88	165.4

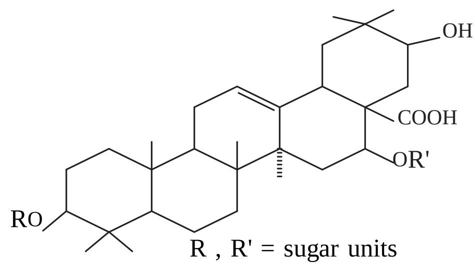

Figure 2. Structure of saponin extracted from shikakai.

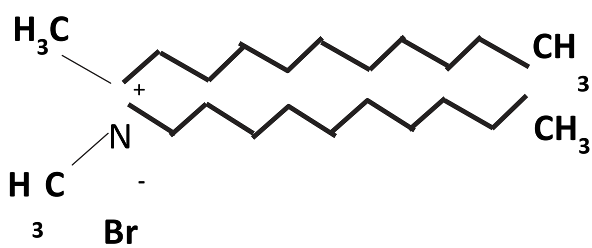

Figure 3. Structure of di-docyl ammonium bromide (DDAB).

Measurement of Surface Tension

With the help of a KYOWA 350 (Japan) surface tensiometer, surface tension of saponin and DDAB solution of different concentration was measured by Wilhelmy plate method.

Preparation of Coal Water Slurry

About 100 ml of CWS samples in concentration range of 58–70% by weight were prepared in distilled water medium with the addition of 1 wt.% of the saponin and DDAB surfactant/additive mixtures. The procedure to prepare CWS was standardized for all tested samples.

Rheological Measurement

Rheological study of CWS was conducted using a microprocessor based rheometer manufactured by Thermo Scientific Inc. USA with model HAAKE RHEO STRESS 1. The rheometer is equipped with oil-free compressor, temperature control system, computer, and printer. The Cup and Bob sensor system Z was used for the experiment which comprises of a collapsible beaker Z43 with radius 21.7 mm and rotors of Z38 and Z41 with radii 19.01 and 20.71 mm, respectively. The measurement and evaluation of rheological data in the rheometer is controlled by the HAAKE RheoWin software. Slurries prepared at the desired concentrations were subjected to rheological measurements under controlled shear rates (0–300 s^{-1}) and at room temperature of 30°C. Initially cup and rotor distance was set to zero and the cup was filled with approximately 30 ml of slurry and after assembling the rotor and spindle, the experiment was conducted. The rotor was separated from the spindle and the cup was cleaned for repeating the experiment. Cleaned set-up was again used in the subsequent test run. The experiment was repeated twice for each individual slurry sample at a specific solids concentration to ascertain the repeatability of the data. The rheological data were displayed on a computer screen which were recorded on a printer for analysis.

Static Stability

The CWS was prepared at different weight concentrations, i.e. 58%, 62%, 68%, and 70% for sample S-1 in the presence of saponin and DDAB. The slurry samples were placed separately in four 100 ml beakers covered with aluminum foil and left for few days for settling. Stability test was conducted by rod penetration method. Then, a glass rod of 5 mm diameter and 19 g mass was employed to perform penetration test in which the glass rod was inserted into the slurry in every 24 h and the distance travel by the rod was noted down.

Measurement of pH Value

A thermoscientific ORION STAR A211 pH meter was used to measure the pH of slurry. The electrode of the pH meter was dipped in the slurry for 5 h and stirred continuously. The pH value of the slurry was measured at every half an hour interval for a period of five hours. After end of 5 h, the pH of the slurry was found to be stable and considered as the final pH of the slurry. The test was conducted at room temperature and distilled water was used for preparation of slurry.

Determination of Zeta Potential

Zeta potential was measured by Zeta probe 24V (52–60 Hz) T3A, containing 20 wt.% fraction of coal in water. This slurry was stirred for 30 min at 500 rpm and at 25°C to get a homogeneous mixture. One milliliter of the slurry sample was taken for the analysis of zeta potential. Similarly, the zeta potential of coal samples in presence of saponin and DDAB was measured at different concentrations maintaining the above conditions. The experiments were carried out in triplicate and the average values were reported.

Results and Discussion

Surface Tension of Shikakai-Didocyl Ammoniumbromide

The stability of a high concentrated CWS largely depends upon the nature of additives to be get adsorbed onto the surface of coal. This results in reduction of coal agglomeration and helps in slurry stabilization. The behavior of the mixed additive systems i.e. saponin from shikakai and DDAB solution has been observed from surface tension measurement of the mixed solutions at different increased proportion of DDAB concentration.

Figure 4 shows the effect of variable concentration of additive mixture on surface tension value. From the plot it is observed that the surface tension decreases with increase in DDAB concentration in the saponin. In the absence of commercial additive DDAB, one monolayer film of saponin additive is covered on the air–water interface. On adding DDAB solution, surface tension of the system decreases because of the partitioning of DDAB to the interface till a compact mixed monolayer is formed at the air–water interface. It is indicated from the plot that the surface tension of saponin alone could able achieve a value of 27 mN/m. The surface tension gradually decreases with increasing concentration of DDAB in the additive mixture and remains almost constant beyond a concentration of 5% DDAB. So from this study it can be inferred that the optimum dosage of DDAB in the saponin may be taken as 5% as there is no further decrease in the value of surface tension. Since hydrophobicity is maximum at the minimum value of surface tension, the ratio of (95:5)% of saponin and DDAB concentration was taken for the stabilization of CWS.

Optimum Particle Size (Particle Size vs. Apparent Viscosity)

Particle size and size distribution are two important parameters for the preparation of high concentration minerals/ores suspensions. Variation in the particle size modifies the flow behavior of the suspension and influences the rheological parameters. Therefore, determination of particle size is an important factor during rheological measurement. Mineral slurries with finer particle sizes indicate higher apparent viscosity than with the coarse particles (Buranasrisak and Narasingha 2012). So a combination of fine and coarse fractions of particle sizes i.e. bimodal distribution of the coal sample has been chosen for the present study. The plot of apparent viscosity versus shear rate for the four representative coal samples (S-1, S-2, S-3, and S-4) at a slurry concentration of 62% by weight is

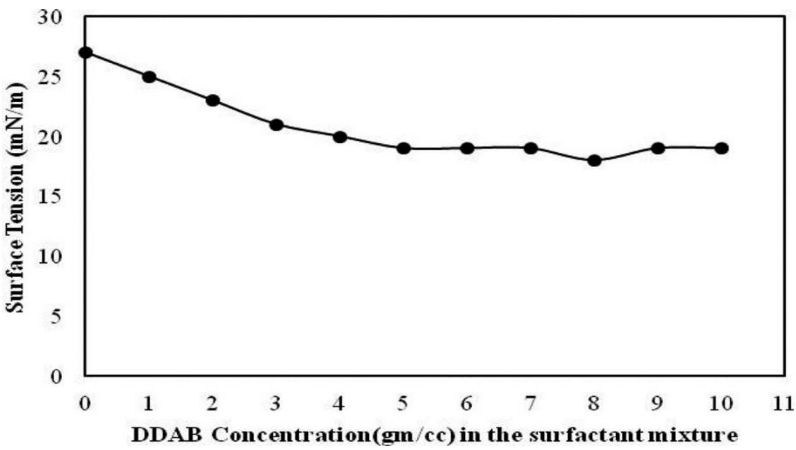

Figure 4. Influence of DDAB concentration in saponin-DDAB mixture on surface tension.

shown in Figure 5. It is observed from Figure 5 that the apparent viscosities of the four CWS samples indicated a decreasing trend with increase in shear rate. Among all the samples, S-1 indicated lowest apparent viscosity values in the studied range of shear rates. This may be due to the fact that the coarse particle size contains relatively large voids so as to allow water to flow in the voids and enhance the fluidity of the slurry. So the above particle size is chosen for the rheology of CWS.

Determination of Optimum Coarse to Fine Particle Ratio

Figure 6 represents shear rate and apparent viscosity of sample S-1 with different coarse to fine ratio at a slurry concentration of 62% by weight.

From the graph it is seen that the slurry sample containing coarse to fine ratio 60:40 has less viscosity than those of containing coarse to fine ratio 70:30 and 65:35. This is because with increase in presence of coarse particles in the coarse-fine mixture, the voidage space probably reduces resulting higher resistance for CWS to commence flow. So from rheology point of view, the optimum particle ratio of coarse to fines in sample S-1 may be considered as 60:40.

Effect of Shear Rate on Shear Stress of Coal Water Slurry

Newtonian fluids exhibit a linear relationship between the applied shear stress and the shear rate. Flow is initiated as soon as a shear stress is applied. The linear relationship between the shear stress and shear rate indicates a constant viscosity. Concentrated mineral and ores fines suspensions often display non-Newtonian flow behavior in that they possess a yield stress that must be exceeded before flow can occur. In the present investigation, the flow behavior of CWS were evaluated at different weight concentrations

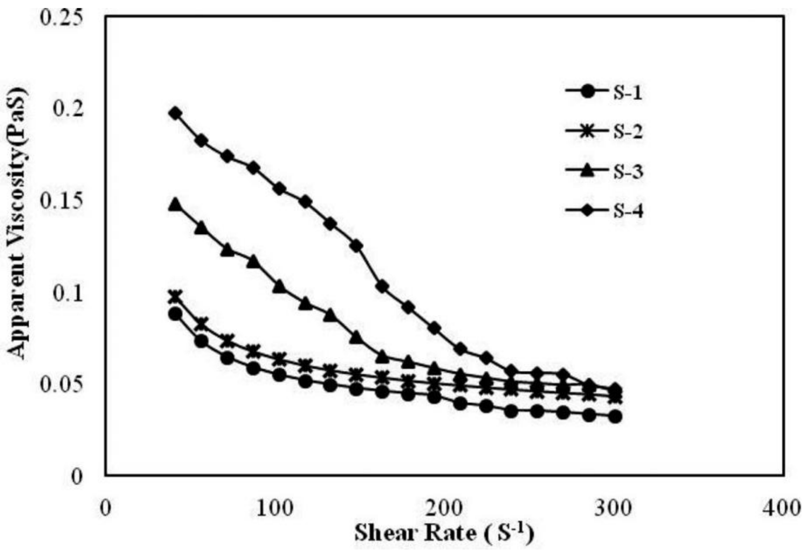

Figure 5. Effect of shear rate on apparent viscosity of coal water slurry for a particular concentration 62 wt.% for four samples S-1, S-2, S-3, and S-4.

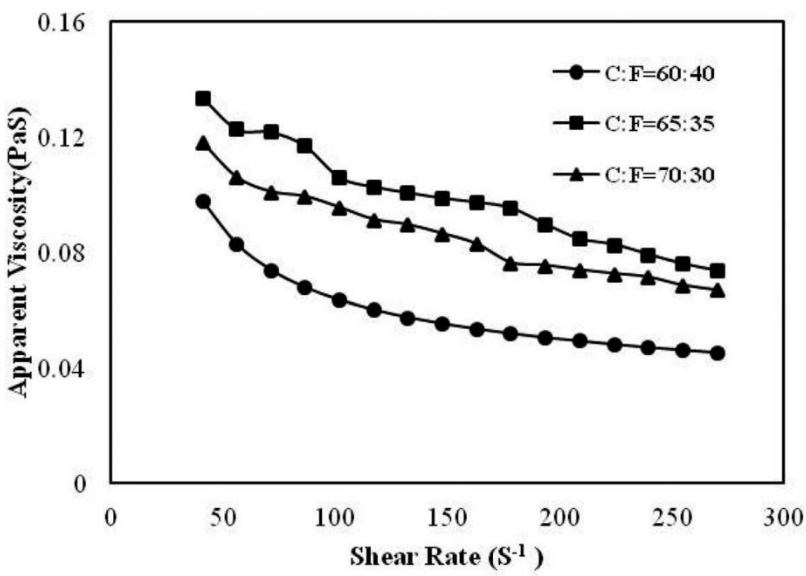

Figure 6. Effect of shear rate on apparent viscosity for 62 wt.% coal loading for sample S-1 at different coarse and fine ratio (C:F) of coal.

i.e. 62%, 68%, and 70% with the variation of shear rate in the presence of additive mixture containing saponin and DDAB.

Figure 7 shows the relationship between shear stress and shear rate at different weight concentration for sample S-1 with a coarse to fines ratio of 60:40. It is seen from the figure that the shear stress increased with increase in shear rate for all concentrations and

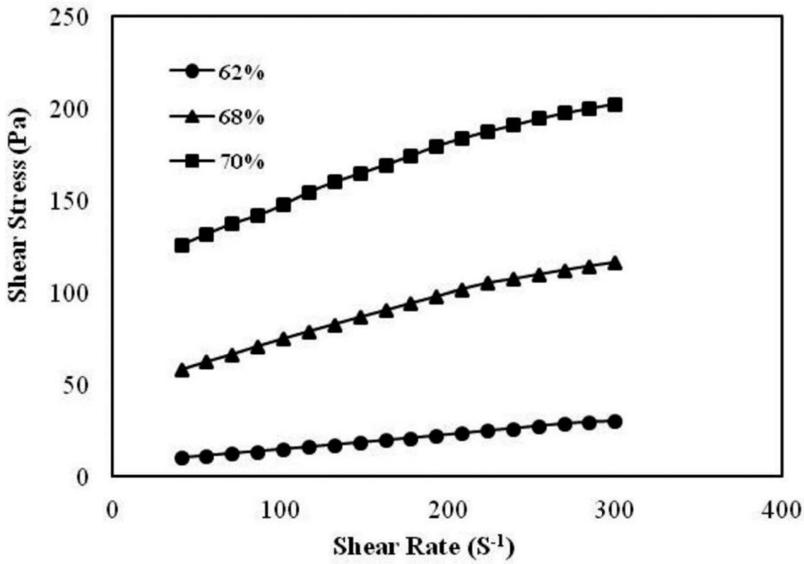

Figure 7. Effect of shear rate on shear stress of coal water slurry at different concentrations for sample S-1 at C:F = 60:40.

indicated non-Newtonian flow behavior with some yield stress (Miller, Laskowski, and Chang 1983). These types of curve with yield stress followed by a linear shear stress – shear rate relationship can be fitted to Bingham Plastic Model. Thus, the constitutive relationship to characterize the flow behavior of CWS may be written as follows:

$\tau = \tau_0 + \eta_p \gamma$

Where, τ = shear stress (Pa)

γ = shear rate (S^{-1})

τ_0 = yield stress (Pa)

η_p = Plastic viscosity (Pas)

The Bingham parameters such as yield stress and apparent viscosity values of sample S-1 at a slurry concentration of 58% by weight with and without additives are presented in Table 4. Also, the measured values of these parameters at higher solids concentration of 62%, 68%, and 70% with the mixture additive are given in Table 4.

It is seen from the table that the presence of saponin and DDAB mixture modified the flow behavior of the CWS. There was a reduction in viscosity as well as yield stress values at a slurry concentration of 58% by weight and the percentage reduction in viscosity and yield stress were found to be 16.5% and 33.2%, respectively. Since, it was not possible to prepare CWS without additives beyond 58% by weight, therefore, the comparison of Bingham parameters at higher solids concentration could not be evaluated. However, the study indicates that the application of additive mixture will be quite beneficial in preparing high concentration CWS and influencing the rheological parameters.

Effect of Solids Concentration on Apparent Viscosity

Figure 8 shows the effect of solids concentration on apparent viscosity for sample S-1 with a coarse to fines ratio of 60:40 in the presence of saponin and DDAB (saponin: DDAB = 95:5).

The apparent viscosity of slurry increases with increase in solids concentration from 62% to 70% by weight. This is because the particle–particle interaction predominates in

ISSN: 2278-4632
Vol-10 Issue-1 January 2020

the concentrated slurry, which results in increasing friction among layers. Similar types of results were observed by some investigator while conducting rheological studies on Indian coal samples (Senapati et al. 2008; Das et al. 2009).

Table 4. Apparent viscosity and yield stress for sample S-1 in the presence of saponin and DDAB.

Weight concentration (*Cw*, %)	Apparent viscosity (Pas) without additive	Yield stress (Pa) without additive	Apparent viscosity (Pas) with additive	Yield stress (Pa) with additive
58	0.0575	2.875	0.048	1.92
62	-	-	0.1261	11.69
68	-	-	0.5885	40.19
70	-	-	1.12	42.35

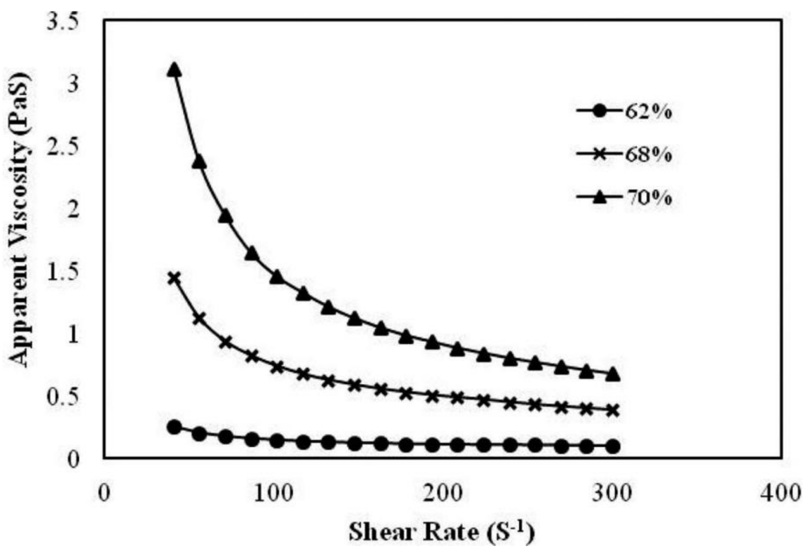

Figure 8. Effect of shear rate on apparent viscosity of coal water slurry for S-1 sample at C:F = 60:40.

Variation of the Additive Ratio at Fixed Coal Concentration

Figure 9 shows the effect of mixture of saponin and DDAB concentration on apparent viscosity of CWS. Keeping the total amount of additive mixture in the slurry as 1 wt.%, the amount of DDAB was varied in the additive mixture from 0% to 10%. From the plot it is seen that minimum viscosity is reached when the additive mixture is in the ratio of saponin: DDAB (95:5) wt.%. This ratio of additive mixture exactly matches with the ratio

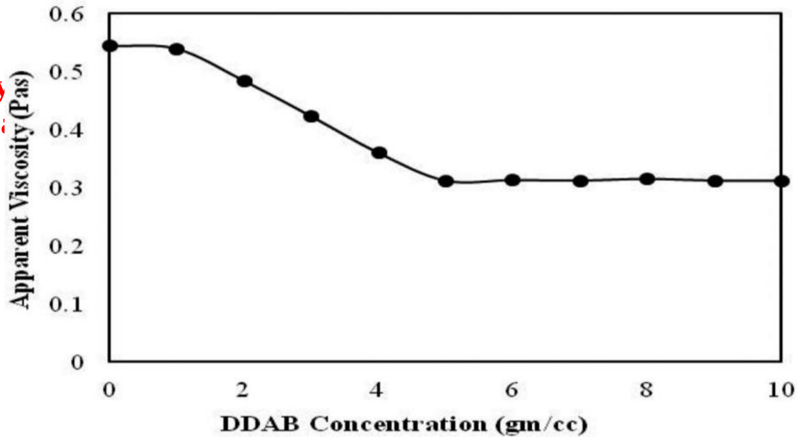

Figure 9. Variation of DDAB concentration in the additive mixture on apparent viscosity of coal water slurry (Sample: S-1, C:F::60:40).

of the mixture in the surface tension measurement. The apparent viscosity gives minimum value because of increase in the electrostatic repulsion among the absorbed additive in the coal interface.

So based on the minimum viscosity as well as minimum surface tension measurement, the optimum value of the additive mixture in CWS preparation may be taken as saponin: DDAB is 95:5 wt.%.

Effect of pH on the Viscosity of Coal Water Slurry

With gradual increase in pH value polar groups like – OH, –COOH attached to the coal surface undergoes ionization and coal surface is covered by intense negative charge. This creates electrostatic repulsion among the dispersed coal particle, which prevents particle-particle association. Thus increase in pH value decreases apparent viscosity of CWS as shown in Figure 10 and no more decreases beyond pH 8.0. Hence a well-dispersed suspension may be obtained at high pH value.

Effect of Additive Concentration with Zeta Potential

Figure 11 shows the variation of zeta potential of CWS in the presence of saponin and mixture of saponin and DDAB affecting zeta potential of CWS system. When only saponin was used, the zeta potential value gradually decreased and indicated a value of – 88 MeV as reported by Das et al. (2013). From the plot it can be seen that addition of 5% of DDAB to 95% of saponin, the zeta potential gradually decreased to –82 MeV. So it is concluded that in the presence of saponin the zeta potential decreases and also when DDAB is added in the saponin the value be negative.

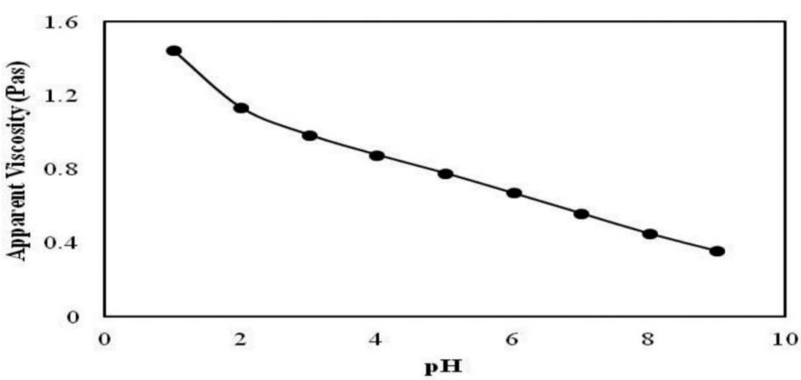

Figure 10. Effect of pH of coal water slurry on apparent viscosity using saponin and DDAB.

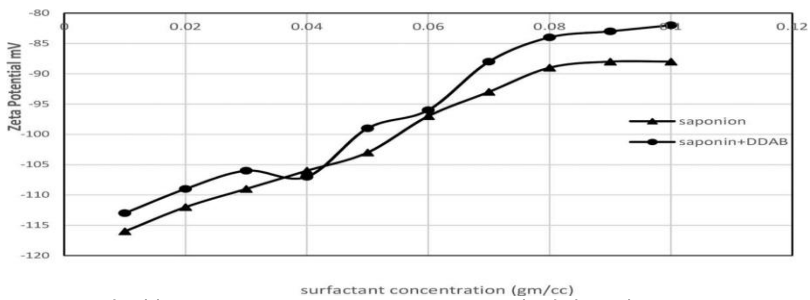

Figure 11. Variation of additive concentration on zeta potential of the solution.

Static Stability Study of Coal Water Slurry

Literature survey indicates that the maximum coal loading and stabilization of CWS can be achieved with dosages of 0.8–1 wt.% of additive (Tiwari et al. 2003; Zhou et al. 2007; Das et al. 2008, 2009, 2010). The application of Shikakai as a selective additive could able to give a maximum coal loading of 64.5% by weight (Das et al. 2013). Accordingly, 1 wt.% of the mixture additive was used which improved coal loading with stabilization of CWS. Table 5 gives comparison of the stability of the slurry in presence of saponin, DDAB, and mixture of saponin and DDAB.

From Table 5 it is observed that static stability of coal in presence of saponin and DDAB alone were found to be 23 days and 24 days, respectively. However, the static stability of CWS improved and was determined to be 29 days when the mixture of saponin and DDAB was used for preparation of CWS.

Mechanism of Stabilization for Coal Water Slurry

Mechanism of CWS can be explained by various phenomena such as Hydrogen bonding (Law and Kunze 1966; Synder 1968; Zhang, Liao, and Shi 2005), hydrophobic interaction (Dick, Fuersteanau, and Healy 1971; Giles, D'Silva, and Easton 1974; Wakamatsu and Fuersteanau 1968), ion-pairing (Zhang, Liao, and Shi 2005; Law and Kunze 1966), and ion exchange (Zhang, Liao, and Shi 2005; Law and Kunze 1966). Since zeta potential of the slurry is decreasing in the mixed surfactant system, mechanism of coal water interaction can be explained on the basis of steric effect (Das et al. 2009;, 2010) instead of electrostatic interaction. Since surface activity of the mixed surfactant system is at 95:5 (w/w) of saponin and DDAB, maximum amount of additive mixture is supposed to be adsorbed at this ratio. Due to micelle formation, there is no more partition of DDAB or saponin to coal surface (Kilau and Voltz 1991).

Table 5. Stability of coal water slurries in days with additives at C_W = 70%.

Additives	Days
Saponin	23
DDAB	24
Saponin+DDAB	29

Figure 12 shows linking of DDAB–saponin at coal–water interface. Saponin consists of two parts, one is aglycone (tri-terpenoid skeleton) called hydrophobic and other is sugar residue called hydrophilic which is also known as glycone part. Aglycone, tri-terpenoid skeleton is attached to the coal surface and dodecyl chain (hydrocarbon) of DDAB through it's lower face and upper face, respectively. Thus a strong hydrophobic bonding

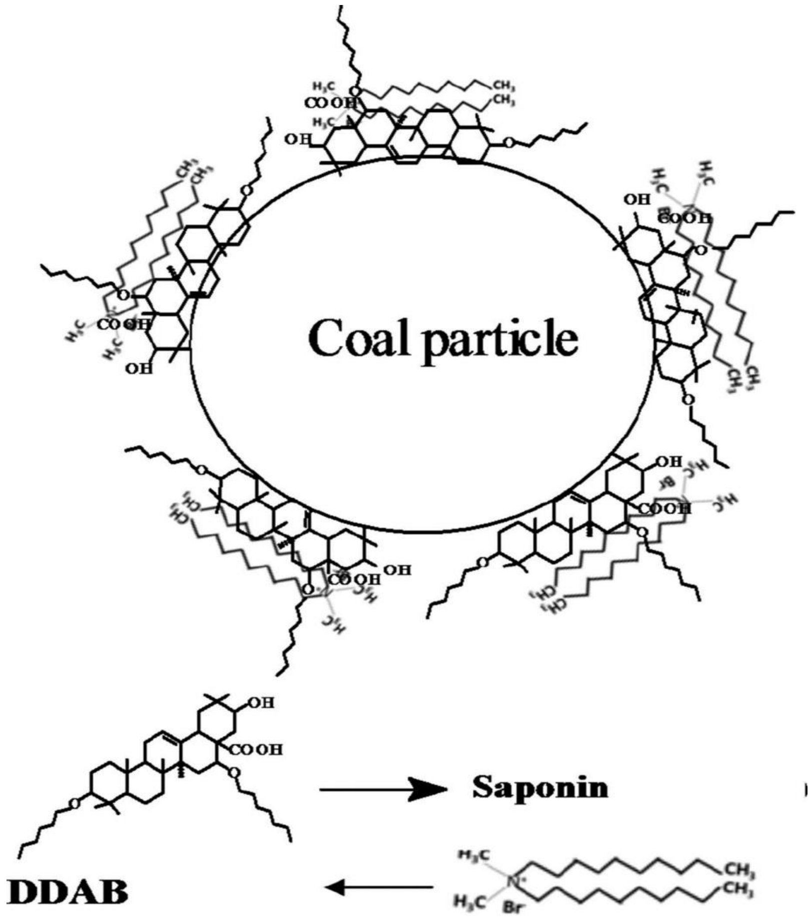

Figure 12. Structure of DDAB-saponin at the coal–water interface.

takes place between DDAB and saponin because of the same no of carbon atoms in the upper face of aglycone part (triterpene skeleton) of saponin and DDAB. This results a better stabilization of CWS (Das et al. 2013).

Conclusions

The salient features of the present experimental results are highlighted below:

(i) It is quite feasible to prepare CWS by using mixture natural and synthetic additives.
(ii) The ratio of Saponin and DDAB in the additive mixture was optimized by measuring surface tension of the solution.
(iii) The flow behavior of CWS with and without additives may be characterized by non-Newtonian Bingham plastic model.
(iv) Bimodal distribution coal sample in a fixed weight ratio of coarse to fines ratio of 60:40 improved the coal loading as well as reduced the viscosity in CWS.
(v) Wet ability of coal occurs because of large number of –OH group available in saponin molecule and electrophilic ammonium ion in DDAB.
(vi) Zeta potential of CWS displays a downward trend in the presence of additive confirming the role of steric stabilization in dispersion of CWS.
(vii) Apparent viscosity of the slurry gradually decreases with increase in pH of CWS.
(viii) Since the additive used for the study was free of sulfate and sulfonate group,

therefore the emission of harmful gases like SO_x, NO_x, etc. may be ruled out amid the combustion of CWS.

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