EXPERIMENTAL INVESTIGATION OF RHEOLOGICAL CHARACTERISTICS OF COKING COAL SLURRY AND NUMERICAL ANALYSIS OF DIFFUSIVITY IN SOLID LIQUID SLURRY

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by

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Submitted

in fulfilment of the requirements of the degree of Doctor of Philosophy

To the

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Dedicated to my beloved mother

Mrs. Neeraj Singh

Certificate

This is to certify that the thesis entitled "**Experimental Investigation of Rheological Characteristics of Coking Coal Slurry and Numerical Analysis of Diffusivity in Solid Liquid Slurry**" being submitted by **Mr. Himanshu Pratap Singh** to the **Indian Institute of Technology Delhi** for the award of the degree of **Doctor of Philosophy** in civil engineering is a bonafide record of research work carried out by him under my supervision and guidance. The thesis work, in my opinion, has reached the requisite standard fulfilling the requirements for the degree of Doctor of Philosophy. The research reports and results presented in this thesis have not been submitted, in part or full, to any other University or Institute for the award of any degree or diploma.

(Dr. Deo Raj Kaushal)

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Himanshu Pratap Singh

Abstract

Coking coal is an important ingredient used in steel manufacturing industries. It is the type of coal which possess very low ash and Sulphur content and when exposed to very high temperature in the absence of air, a highly carbon rich compound i.e., coke is prepared. This coke is used as a strong reducing agent for the metals or metal oxide in the process of manufacturing of steel. Hence, the transportation of coking coal is required on large scale. In the present study, rheological aspects of the coking coal and water slurry (CCWS) are studied experimentally under different circumstances and a modified model for the coefficient of particle diffusivity for solid-liquid slurry flows is proposed.

Rheological study on CCWS is performed using rotational rheometer for lower mass concentrations 20%, 25% and 30% and for higher mass concentrations 50%, 55%, 60% and 65%. At lower shear rate range shear thickening behaviour is exhibited by the CCWS up to 50% mass concentration and then it starts changing to shear thinning behaviour for higher concentrations. But in case of higher shear rate range, shear thickening behaviour is exhibited up to 60% mass concentration. The slurry behaviour has also been observed to be varying with different time intervals of soaking of CCWS. Sedimentation analysis of CCWS is performed and it was found that the settling of solids is taking place as long as up to 48 hours. Hydrophobic nature of the coking coal particles is clearly visible on the top surface of CCWS after mixing the solids with water in the cylindrical flask. Further absorption of water by the particles take place and the highly porous structure of the coking coal particles influence the solid liquid interaction with time. Hence, the rheology of CCWS is also determined at different time intervals (0 hours, 24 hours, 28 hours, 48 hours, and 72 hours) for the same sample kept in controlled environment. Rheological behaviour of the slurry is found to be changing significantly and the apparent viscosity increase with increasing soaking time interval. Practically such situations can be observed while the non-functioning of pipelines, storage of slurries in sharp vertical bends and slurry storage at different terminals. Slurry mixture follows the Herschel Bulkley model of rheology, for which all the rheological parameters are determined and analysed. Correlations for all the parameters are formulated for which the predicted values are found to be in close agreement with the experimental data.

Transportation of highly concentrated slurries in the industry is required to save time and to make the system more cost effective. Hence, the chemical additives are used to keep pumping power consumption less and mixed with the slurry which helps in reducing the slurry viscosity. In

the present study, chemical additive Calcium Hydroxide is used as additive for the CCWS. Rheological tests are conducted on the samples of CCWS with mass concentrations 20% and 25%. Tests are performed by adding the Calcium Hydroxide to the samples at mass concentrations 0.05%, 0.1%, 0.5%, 1%, 1.5% and 2%. A significant change in the apparent viscosity of the CCWS is observed with the addition of Calcium Hydroxide to the CCWS. CCWS samples with Calcium Hydroxide are also tested at different soaking time intervals i.e., 0 hours, 24 hours, 28 hours, 48 hours and 72 hours. Shear stress and the apparent viscosity decreases with the addition of Calcium Hydroxide to the CCWS. For 20% mass concentration at lower shear rates, at 48 hours and 72 hours soaking time intervals, the shear stresses are found to be minimum for 0.5% additive concentration and for 0 hours, 24 hours and 28 hours soaking time intervals, the shear stresses are found to be minimum for 2% additive concentration. For higher shear rates, at 48 hours and 72 hours soaking time intervals, the shear stresses are found to be minimum for 1.5% additive concentration and for 0 hours, 24 hours and 28 hours soaking time intervals, the shear stresses are found to be minimum for 2% additive concentration. For 25% mass concentration at lower shear rates, at 48 hours and 72 hours soaking time intervals, the shear stresses are found to be minimum for 1.5% additive concentration and for 0 hours, 24 hours and 28 hours soaking time intervals, the shear stresses are found to be minimum for 1% additive concentration. For higher shear rates, at 28 hours, 48 hours and 72 hours soaking time intervals, the shear stresses are found to be minimum for 0.5% additive concentration and for 0 hours and 24 hours soaking time intervals, the shear stresses are found to be minimum for 0.05% additive concentration. Numerical analysis for experimental results obtained is performed and the correlations for the rheological parameters are proposed agreeing well with the experimental data.

In order to optimise and estimate the pressure drop for the slurry flow through pipelines, it is required to model the slurry flows and estimate the concentration profiles more accurately. Many researchers have adopted various methods for predicting concentration profiles in slurry pipeline flows till now. Kaushal and Tomita (2002) found that the particle diffusivity is a varying parameter which depends upon the total efflux concentration and the static settled concentration as well. They obtained single value of the coefficient of particle diffusivity for each of the concentration profile. This model assumes the varying coefficient of particle diffusivity in place of constant particle diffusivity as considered by Karabelas (1977). The modelling approach is based on the diffusion model and the assumption that gravitational force of the settling particles in a slurry flow is balanced by the upward movement of the particles due to particle diffusion. In the present study, a numerical model for the coefficient of particle diffusivity proposed by Kaushal and Tomita (2002) is modified after taking into consideration the variation of particle diffusivity along the

vertical distance from the pipe bottom. Experimental data provided by Matousek et al. (2014) and Krupicka and Matousek (2014) has been used to optimise the coefficient of particle diffusivity at different locations corresponding to the respective particle concentration. Matousek et al. (2014) and Krupicka and Matousek (2014) performed experiments on slurries prepared with glass beads flowing through pipeline and the concentration profiles measured using a highly advanced set up of γ-ray densitometer for different flow parameters. Values obtained for the coefficient of particle diffusivity are found to be increasing with the increase in vertical distance from the pipe bottom. Larger particles possess higher values of particle diffusivity. A mathematical expression for the coefficient of particle diffusivity is proposed which is a function of the vertical distance from the pipe bottom, pipe diameter, total efflux concentration and the static settled concentration. The results from the proposed model are found to be closer to the experimental data as compared to that of the Kaushal and Tomita (2002) model. Standard deviations of concentration profiles from the experimental data for the proposed model are also reduced as compared to that of the Kaushal and Tomita (2002) model.

संक्षेप

कोकिंग कोल इस्पात निर्माण उद्योगों में इस्तेमाल होने वाला एक महत्वपूर्ण घटक है। यह कोयले का प्रकार है जिसमें बहुत कम राख और सल्फर की मात्रा होती है और हवा की अनुपस्थिति में बहुत उच्च तापमान के संपर्क में आने पर अत्यधिक कार्बन समृद्ध यौगिक यानी कोक तैयार किया जाता है। स्टील के निर्माण की प्रक्रिया में इस कोक का उपयोग धातुओं या धातु ऑक्साइड के लिए एक मजबूत अपचायक के रूप में किया जाता है। इसलिए, कोकिंग कोल का परिवहन बड़े पैमाने पर आवश्यक है। वर्तमान अध्ययन में, कोकिंग कोयले और पानी के घोल (सीसीडब्ल्यूएस) के रियोलॉजिकल पहलुओं का विभिन्न परिस्थितियों में प्रयोगात्मक रूप से अध्ययन किया जाता है और ठोस-तरल घोल प्रवाह के लिए कण प्रसार के गुणांक के लिए एक संशोधित मॉडल प्रस्तावित है।

सीसीडब्ल्यूएस पर रियोलॉजिकल अध्ययन 20%, 25% और 30% कम द्रव्यमान सांद्रता के लिए और उच्च द्रव्यमान सांद्रता 50%, 55%, 60% और 65% के लिए घूर्णी रियोमीटर का उपयोग करके किया जाता है। कम कतरनी दर सीमा पर कतरनी मोटा होना व्यवहार सीसीडब्ल्यूएस द्वारा 50% द्रव्यमान एकाग्रता तक प्रदर्शित किया जाता है और फिर यह उच्च सांद्रता के लिए कतरनी के पतले व्यवहार में बदलना शुरू कर देता है। लेकिन उच्च कतरनी दर सीमा के मामले में, कतरनी मोटा होना व्यवहार 60% द्रव्यमान एकाग्रता तक प्रदर्शित होता है। सीसीडब्ल्यूएस को भिगोने के अलग-अलग समय अंतरालों के साथ घोल का व्यवहार भी अलग-अलग देखा गया है। CCWS का अवसादन विश्लेषण किया जाता है और यह पाया गया कि ठोस पदार्थों का निपटान 48 घंटे तक हो रहा है। कोकिंग कोल कणों की हाइड्रोफोबिक प्रकृति बेलनाकार फ्लास्क में पानी के साथ ठोस पदार्थों को मिलाने के बाद सीसीडब्ल्यूएस की ऊपरी सतह पर स्पष्ट रूप से दिखाई देती है। कणों द्वारा पानी का और अधिक अवशोषण होता है और कोकिंग कोयले के कणों की अत्यधिक झरझरा संरचना समय के साथ ठोस तरल संपर्क को प्रभावित करती है। इसलिए, नियंत्रित वातावरण में रखे गए एक ही नमूने के लिए सीसीडब्ल्यूएस की रियोलॉजी भी अलग-अलग समय अंतराल (0 घंटे, 24 घंटे, 28 घंटे, 48 घंटे और 72 घंटे) पर निर्धारित की जाती है। स्लरी के रियोलॉजिकल व्यवहार में काफी बदलाव देखा गया है और भिगोने के समय अंतराल में वृद्धि के साथ स्पष्ट चिपचिपाहट बढ़ जाती है। व्यावहारिक रूप से ऐसी स्थितियों को देखा जा सकता है जब पाइपलाइनों का काम न करना, तेज ऊर्ध्वाधर मोड़ों में स्लरी का भंडारण और विभिन्न टर्मिनलों पर स्लरी भंडारण। गारा मिश्रण रियोलॉजी के हर्शल बल्कली मॉडल का अनुसरण करता है, जिसके लिए सभी रियोलॉजिकल पैरामीटर निर्धारित और विश्लेषण किए जाते हैं। उन सभी मापदंडों के लिए सहसंबंध तैयार किए गए हैं जिनके लिए अनुमानित मान प्रयोगात्मक डेटा के साथ निकट समझौते में पाए जाते हैं।

समय बचाने और प्रणाली को अधिक लागत प्रभावी बनाने के लिए उद्योग में अत्यधिक केंद्रित स्लरी का परिवहन आवश्यक है। इसलिए, रासायनिक योजक का उपयोग पंपिंग बिजली की खपत को कम रखने और घोल के साथ मिश्रित करने के लिए किया जाता है जो घोल की चिपचिपाहट को कम करने में मदद करता है। वर्तमान अध्ययन में, रासायनिक योज्य कैल्शियम हाइड्रॉक्साइड का उपयोग CCWS के लिए योज्य के रूप में किया जाता है। 20% और 25% द्रव्यमान सांद्रता वाले CCWS के नमूनों पर रियोलॉजिकल परीक्षण किए जाते हैं। बड़े पैमाने पर सांद्रता 0.05%, 0.1%, 0.5%, 1%, 1.5% और 2% पर नमूनों में कैल्शियम हाइड्रॉक्साइड जोड़कर टेस्ट किए जाते हैं। CCWS की स्पष्ट चिपचिपाहट में एक महत्वपूर्ण परिवर्तन CCWS में कैल्शियम हाइड्रॉक्साइड के अतिरिक्त के साथ देखा गया है। कैल्शियम हाइड्रॉक्साइड के साथ सीसीडब्ल्यूएस के नमूनों का भी अलग-अलग भिगोने के समय अंतरालों यानी 0 घंटे, 24 घंटे, 28 घंटे, 48 घंटे और 72 घंटे पर परीक्षण किया जाता है। सीसीडब्ल्यूएस में कैल्शियम हाइड्रॉक्साइड को शामिल करने से कतरनी तनाव और स्पष्ट चिपचिपाहट कम हो जाती है। कम अपरूपण दरों पर 20% द्रव्यमान सांद्रता के लिए, 48 घंटे और 72 घंटे के समय अंतराल पर, कतरनी तनाव न्यूनतम 0.5% योगात्मक एकाग्रता के लिए और 0 घंटे, 24 घंटे और 28 घंटे के समय अंतराल के लिए, कतरनी तनाव पाया जाता है। 2% योज्य सांद्रता के लिए न्यूनतम पाए जाते हैं। उच्च कतरनी दरों के लिए, 48 घंटे और 72 घंटे के समय अंतराल पर, कतरनी तनाव 1.5% योगात्मक एकाग्रता के लिए न्यूनतम पाया जाता है और 0 घंटे, 24 घंटे और 28 घंटे के समय अंतराल के लिए, कतरनी तनाव न्यूनतम पाया जाता है। 2% योगात्मक एकाग्रता के लिए। कम कतरनी दर पर 25% द्रव्यमान एकाग्रता के लिए, 48 घंटे और 72 घंटे के समय अंतराल पर, कतरनी तनाव न्यूनतम 1.5% योगात्मक एकाग्रता के लिए पाया जाता है और 0 घंटे, 24 घंटे और 28 घंटे के समय अंतराल के लिए, कतरनी तनाव 1% योज्य सांद्रता के लिए न्यूनतम पाए जाते हैं। उच्च कतरनी दरों के लिए, 28 घंटे, 48 घंटे और 72 घंटे के समय अंतराल पर, कतरनी तनाव 0.5% योगात्मक एकाग्रता के लिए न्यूनतम पाया जाता है और 0 घंटे और 24 घंटे के समय अंतराल के लिए, कतरनी तनाव न्यूनतम पाया जाता है। 0.05% योज्य एकाग्रता के लिए। प्राप्त प्रयोगात्मक परिणामों के लिए संख्यात्मक विश्लेषण किया जाता है और प्रयोगात्मक डेटा के साथ अच्छी तरह से सहमत होने के लिए रियोलॉजिकल पैरामीटर के लिए सहसंबंध प्रस्तावित किए जाते हैं।

पाइपलाइनों के माध्यम से गारा प्रवाह के लिए दबाव ड्रॉप का अनुकूलन और अनुमान लगाने के लिए, गारा प्रवाह को मॉडल करना और एकाग्रता प्रोफाइल का अधिक सटीक अनुमान लगाना आवश्यक है। कई शोधकर्ताओं ने अब तक गारा पाइपलाइन प्रवाह में एकाग्रता प्रोफाइल की भविष्यवाणी करने के लिए विभिन्न तरीकों को अपनाया है। कौशल और टोमिता (2002) ने पाया कि कण प्रसार एक अलग-अलग पैरामीटर है जो कुल प्रवाह एकाग्रता और स्थिर बसे हुए एकाग्रता पर भी निर्भर करता है। उन्होंने प्रत्येक सांद्रता प्रोफ़ाइल के लिए कण प्रसार के गुणांक का एकल मान प्राप्त किया। यह मॉडल कारबेलस (1977)

द्वारा माने गए निरंतर कण प्रसार के स्थान पर कण प्रसार के भिन्न गुणांक को मानता है। मॉडलिंग दृष्टिकोण प्रसार मॉडल पर आधारित है और यह धारणा है कि घोल प्रवाह में बसने वाले कणों का गुरुत्वाकर्षण बल कण प्रसार के कारण कणों के ऊपर की ओर गति से संतुलित होता है। वर्तमान अध्ययन में, कौशल और टोमिता (2002) द्वारा प्रस्तावित कण प्रसार के गुणांक के लिए एक संख्यात्मक मॉडल को पाइप तल से ऊर्ध्वाधर दूरी के साथ कण प्रसार की भिन्नता को ध्यान में रखते हुए संशोधित किया गया है। मैटौसेक एट अल द्वारा प्रदान किया गया प्रायोगिक डेटा। (2014) और कृपिका और मैटौसेक (2014) का उपयोग संबंधित कण एकाग्रता के अनुरूप विभिन्न स्थानों पर कण प्रसार के गुणांक को अनुकूलित करने के लिए किया गया है। माटोसक एट अल। (2014) और कृपिका और माटोसेक (2014) ने पाइपलाइन के माध्यम से बहने वाले कांच के मोतियों के साथ तैयार स्लरी पर प्रयोग किए और विभिन्न प्रवाह मापदंडों के लिए γ-रे डेंसिटोमीटर के अत्यधिक उन्नत सेट का उपयोग करके मापा गया एकाग्रता प्रोफाइल। पाइप के तल से लंबवत दूरी में वृद्धि के साथ कण प्रसार के गुणांक के लिए प्राप्त मान बढ़ते पाए जाते हैं। बड़े कणों में कण विसारकता के उच्च मूल्य होते हैं। कण विसरणशीलता के गुणांक के लिए एक गणितीय अभिव्यक्ति प्रस्तावित है जो पाइप के तल से ऊर्ध्वाधर दूरी, पाइप व्यास, कुल प्रवाह एकाग्रता और स्थिर स्थिर एकाग्रता का एक कार्य है। प्रस्तावित मॉडल के परिणाम कौशल और तोमिता (2002) मॉडल की तुलना में प्रायोगिक डेटा के करीब पाए गए हैं। कौशल और टोमिता (2002) मॉडल की तुलना में प्रस्तावित मॉडल के लिए प्रायोगिक डेटा से एकाग्रता प्रोफाइल के मानक विचलन भी कम किए गए हैं।

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Nomenclature

List of symbols

C_D	Drag coefficient
C_{vf}	Total efflux solid volume concentration
C_{wss}	Static settled concentration of slurry (by weight)
C_{vss}	Static settled concentration of slurry (by volume)
C_v	Volumetric concentration of solids in the slurry
C_w	Mass concentration of solids in the slurry
c_{pi}	Predicted volumetric concentration of the jth size fraction
c_{mi}	Measured volumetric concentration of the jth size fraction
d_j	Mean diameter of the jth size fraction (µm)
d_{50}	Particle mean diameter (µm)
D	Pipe diameter (m)
ε/D	Relative pipe roughness
f	Darcy-Weisbach friction factor
f_L	Friction factor for laminar flow
f_T	Friction factor for turbulent flow
g	Acceleration due to gravity (m/s^2)
G	Specific gravity of solids
He	Hedstrom Number
i_m	Pressure head gradient for the slurry mixture
i_v	Pressure head gradient due to vehicle
K	Flow consistency index ($Pa.s^n$)
K_p	Power law Flow consistency index ($Pa.s^n$)
K_s	Sisko (1958) Flow consistency index ($Pa.s^n$)
L	Pipe length (m)
n	Flow behaviour index

R	Radius of the pipeline
Re	Reynolds number
Re_d	Particle Reynolds number
Re_m	Slurry Reynolds number
Re_{mod}	Modified Reynolds number
V_{lm}	Volume of water in settling slurry (ml)
V_{mi}	Volume of settled slurry at any given time (ml)
V_m	Initial volume of slurry (ml)
V_1	Volume of water taken (ml)
V	Velocity of flow (m/s)
v_c	Critical flow velocity (m/s)
m_s	Mass of solid particles in slurry (gm)
m_w	Mass of water in slurry (gm)
w_{jo}	Unhindered settling velocity of mean diameter of the j^{th} size fraction (m/s)
y	Depth of flow (m)
$\Delta p/l$	Pressure gradient

Greek Symbols

β	Dimensionless particle diffusivity
τ	Shear stress (Pa)
ξ	Dimensionless eddy diffusivity
τ_0	Yield stress (Pa)
τ_0	Casson (1958) yield stresss
ε_s	Particle diffusivity
ε_l	Liquid diffusivity
ρ_l	Density of carrier fluid (Kg/m^3)
ρ_m	Density of slurry (Kg/m^3)
ρ_s	Density of solids in suspension (Kg/m^3)

μ	Apparent Viscosity (Pa.s)
η_b	Bingham plastic viscosity of solid suspension (Pa.s)
η_s	Sisko (1958) viscosity (Pa.s)
η_s	Casson (1958) viscosity (Pa.s)
γ	Shear rate (s^{-1})
δ	Boundary layer thickness (m)

Abbreviations

CCWS	Coking coal and water slurry
HL	Hydrated Lime
SD	Standard Deviation