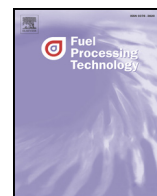




Contents lists available at ScienceDirect

Fuel Processing Technology

journal homepage: www.elsevier.com/locate/fuproc

Research article

Effects of organic liquids on coking properties of a higher-inert Western Canadian coal

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ARTICLE INFO

Article history:

Received 10 January 2016

Received in revised form 13 June 2016

Accepted 16 June 2016

Available online xxxx

Keywords:

Organic liquid

Coke quality

Coal property

Carbonization

Storage time

ABSTRACT

The effect of organic liquids (white spirit, perchloroethylene and methylene bromide) typically used in float/sink gravity separations (specific gravity 1.4–1.8) on the fundamental properties of a higher-inert Western Canadian metallurgical coal of mvb rank (Romax 1.22) and its resultant coking ability were examined. Over a 6-month period, untreated (control) and treated coal were characterized using a wide range of analyses including elemental chemistry, FTIR spectroscopy, alkali extraction via light transmittance test and thermal rheology, specifically fluidity, Dilatation, FSI and Caking Index G. Exposure to organic liquids was found to have a minor effect on coal chemistry (Ultimate) and oxidation level (Alkali Extraction via Light Transmission Test and FTIR-Spectroscopy). Trends of H/C, O/C, Light Transmittance and FTIR absorbance spectra versus storage time, following initial 1 h exposure to the organic liquids of varying specific gravities, revealed only minor changes, within the scatter/accuracy of the measurements. However, several coal plastic properties were decreased significantly including Gieseler Maximum Fluidity, Dilatation and G Caking Index. Of these, most affected was Maximum Fluidity which underwent an immediate and dramatic decrease of over 80% following organic liquids treatment. Other rheology indicators including FSI and Sapozhnikov were significantly less sensitive in detecting initial stages of degradation in plastic properties. The coking ability of both the untreated and treated coal in perchloroethylene (PCE) organic liquid, s.g. 1.6, after storage for 1 and 6 months was assessed through carbonization trials in both a small-scale sole-heated oven (12.5 kg capacity) and in a pilot-scale movable wall oven (350 kg capacity) at CanmetENERGY Carbonization facility, Ottawa, Canada. The treated samples had appreciably poorer quality ambient (ASTM, IRSID, JIS tumbler tests) and hot (CSR, CRI) coke strength and modified coke structure. Coke ASTM strength for untreated and treated coal at time 1 month was respectively decreased from 62 down to 57 for Stability and from 72 to 67 for Hardness. Similarly, Coke CSR and CRI for untreated and treated coal at time 1 month was respectively decreased from 74 down to 58 and increased from 21 to 29. The treated sample resulted in a lower coke yield, 71%, in comparison with 78% for the untreated sample, produced coke of smaller mean size, 46 mm, than the untreated sample, 53 mm, and generated an appreciably higher fraction of fines (-12.5 mm), 21%, than untreated sample, 4%. The coke textures from the untreated coal consist of higher carbon forms, mosaic and flow in medium size, compared to those from s.g. 1.6 treated coal of lower carbon forms, very fine and fine mosaic, which renders the coke more reactive to CO₂ gasification during CSR test. The higher effective coking rank, 1.2, and Coke Mosaic Size Index, 2.4, of untreated sample relative to that of treated one, 1.1 and 2.2, respectively, supports the better coke quality of the untreated sample. Extension of storage time to 6 months resulted in negligible changes in coke quality showing that the greatest changes occurred within the first month following exposure to organic liquids. Comparison of 1 month untreated and PCE treated mid coking rank Western Canadian (Romax 1.22) and Australian (Romax 1.17) coals show both to undergo comparable decrease in fluidity (50–60%) post treatment with PCE although only the Western Canadian coal leads to lower dilatation. Coke yield is also decreased for the Western Canadian coal and essentially maintained for the Australian coal. Both CSR and CRI are affected negatively and more so for the Western Canadian coal, 16 point drop in CSR and 8 point increase in CRI, relative to the Australian coal, 3 point drop in CSR and 4 point increase in CRI.

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1. Introduction

During the exploration phase of coal mine development, the evaluation of coal and coke quality is often determined using smaller mass samples collected from drill cores. Drilling is the least expensive method of obtaining representative coal seam samples when compared to developing test pits or adits. If a larger bulk sample is required, it is sometimes possible to use several 6 in. drill program cores. However, depending on the thickness of the seam, even this may be cost prohibitive as a large number of drill holes would need to be used to collect the required large coal mass – i.e. several tonnes.

Clean coal samples for quality testing are normally prepared by screening out coarser fractions while the finest fraction is cleaned by froth flotation. The coarser coal is then cleaned using mixtures of organic liquids (i.e. white spirit, perchloroethylene (PCE) and methylene bromide) in a float-and-sink process. During the float-and-sink procedure, the coal sample is separated at relative densities (specific gravity, s.g.) – i.e. white spirit/PCE for 1.4 s.g., PCE for 1.6 s.g. and PCE/methylene bromide for 1.8 s.g. – that produce clean coal samples at different ash contents typical of what would be produced in a commercial coal washing plant. Project economics are based on the results of the float-sink testing – including information on the yield of clean coal as well as the quality of the cleaned coal and resulting coke. The clean coal coking characteristics for a metallurgical coal deposit are imperative in evaluating project economics (i.e. expected price for the clean coal). Ensuring these coal/coking properties are correctly assessed from drill core samples is critical to correctly evaluate project economics. For many years there has been concern that the organic chemicals used in the float-sink analysis may impact the coking quality results of the clean coal samples. The purpose of this paper is to investigate coke quality issues in treating a higher-inert western Canadian coal sample with organic liquids.

1.1. Organic chemicals and float-sink coal

For years the primary concern in the handling and use of organic liquids such as perchloroethylene (PCE) was the safety risks associated with human exposure. PCE is a known carcinogen and poses a safety hazard for laboratory operators. However, a number of observations about how PCE may impact coal sample coking quality have also been noted.

In 2010 Campbell at ALS Coal Technology, Australia found that organic liquids could interfere with the properties of interest for a coal producer or end user [1]. That same year, Iveson and Galvin completed an ACARP study (C17051) which comprehensively examined the effects of organic liquids on coking properties of coal [2,3]. They concluded that PCE, a chemical that is commonly used in the sink-and-float analysis, had, on one hand, a negative effect on the coking properties of lower rank and lower fluidity coking coals but on the other hand, a negligible or possibly even a small positive effect on CRI and CSR of coals with relatively good initial coking properties. The latter observation pertaining to a positive impact of PCE on coke quality was actually reported earlier by DuBroff et al. at Inland Steel, USA [4]. Their 1985 patent outlined a process for improving the quality of some metallurgical coke resulting from coals with high inert content, which had produced coke of lower than expected stability when compared to the coal rank. They studied several medium-volatile bituminous coal samples which had been soaked and agitated in a PCE bath prior to carbonization. For some of the coals, the resultant coke showed improved stability index, increased hardness index, decreased reactivity and increased tumble strength. It was also found that the carbonization time was decreased. The hypothesis was that the PCE reacted with certain macerals in the coal, producing a “solvent induced reaction product” residue on the coal particles that is highly reactive. In some cases, this reaction product could be thought to ‘increase’ the reactive-to-inerts ratio at the coal particle surfaces.

Contrary to what the Inland Steel patent outlines, Iveson and Galvin found that the negative effect of PCE treatment/exposure was shown to be more significant when coal had high inertinite content (>40%). These coals produced lower strength coke as a result of being exposed to PCE. In fact, for coals with high inertinite content, CRI was increased (an adverse effect) by an average of 15% and CSR values decreased by an average of 25% (also an adverse effect) when the coal had been exposed to PCE prior to coking. This effect was more pronounced after the coal had aged for >16 weeks (oxidized). The explanation proposed by Iveson and Galvin was the high porosity of inertinite, namely semi-fusinite and fusinite, enabled greater access of PCE to the interior of the coal particles. The double bonds and chlorine in the PCE could then react with free radicals during the coking process, leading to depressed fluidity and lower coke strength.

The evidence that organic liquids, as discussed previously, affect the coking properties of low fluidity Australian coals implies that Western Canadian coals, known to have moderate fluidity levels, could be affected in a similar way. Canadian geologists have also found that cleaned drill core coal samples often had lower caking/coking properties than bulk or production coal samples. Based on these observations, the Canadian Carbonization Research Association (CCRA) undertook a program to investigate the impact of the organic solvents used in float-sink procedures on the coal and coke properties of a higher-inert Western Canadian coal sample. The objectives of this research were to:

1. Examine the effect of exposing a higher-inert Western Canadian coal to various organic liquids used in typical float-sink procedures on resulting coal and coke quality; and
2. Determine if storage time influences the resulting coal samples and their coke quality.

2. Materials and methods

A suitable drill-core sample was not available for testing from a CCRA member at the time program was undertaken. Consequently a cleaned higher-inert Western Canadian production coal (Romax = 1.22%) was selected as the test coal for this project. A coal of suitable quality was identified from one production train and a bulk sample taken from the port stock-pile where the train was unloaded.

The gross bulk coal sample was split into subsamples for lab and pilot scale oven testing. There were two types of samples consisting of the original coal (control) and samples treated with organic chemicals. Since the bulk coal production sample included the full spectrum of sizes, the subsamples treated in organic chemicals were first screened to remove the froth flotation product since this fraction is not included in drill core sample float-sink preparations. After the coarser fractions were exposed to the organic solvents, the fines fraction was recombined before lab coal quality testing.

Three smaller mass samples would be soaked for 60 min in different fresh organic liquid blends, representing three different specific gravities:

1. Perchloroethylene mixed with white spirit (1.4 s.g.)
2. Perchloroethylene (1.6 s.g.)
3. Perchloroethylene mixed with methylene bromide (1.8 s.g.)

Larger mass samples were prepared for coke oven testing; one was the “control” untreated sample while the other was for treatment with PCE.

For the samples treated in organic liquids, they were air dried in an air-circulating oven at 38 °C (due to the low flash point of the white spirit).

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