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Lignite clean up of magnesium bisulphite pulp mill effluent as a proxy for aqueous discharge from a ligno-cellulosic biorefinery

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ABSTRACT

Pulp and paper mill effluent derived from an acidic magnesium bisulphite process was chosen to model industrial biorefinery process water effluents that are potentially rejected into the environment. This effluent consisted of high colour, organics and phosphorus load. Some treatments that have been reported to deal with organics and colour removal are photocatalysis, oxidation, electrocoagulation, biological treatments and adsorption. Among these, adsorption is still considered to be one of the simplest and economical methods. Activated carbon is probably the most prominent adsorbent applied in wastewater treatment. However, the high costs associated with its activation, regeneration and maintenance have significant drawbacks restricting its use. Lignite, a low rank coal, is a cheap and readily available material that has well known adsorption properties. Here we report on the use of Loy Yang (LY) lignite as an adsorbent and investigate its selectivity for colour, organics and total phosphorus removals from pulp and paper mill effluents. These results suggest the potential of lignite, as a cheap and readily available adsorbent, to be utilized in industrial biorefinery wastewater treatment.

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1. Introduction

With the significant effort dedicated to the transformation of ligno-cellulosic biomass into a combination of biofuels, green chemicals and materials, it is only a matter of time before industrial biorefinery waste process water effluents are rejected into the environment. As sustainability, low carbon footprint, climate change mitigation and other environmental requirements are the major driving forces for the biorefinery, it will be necessary to develop water treatments systems equally environmentally friendly, yet practical and economical. This is the objective of this study.

A Pulp and Paper mill is currently the best known and most common biorefinery. Compounds present in pulp and paper wastewaters include wood polymers and extractives, inorganic fillers, process chemicals and reaction products in the form of suspended solids, colloidal and dissolved matter. Depending on the mill process, hundreds of different organic compounds (e.g., lignins, alcohols, polysaccharide fragments, resin and fatty acids) and inorganic compounds can be found (e.g., Mg_2CO_3, Mg_2SO_4, Mg_2S, MgOH, MgCl, $CaCO_3$ and derivatives, Alum and derivatives) [1,2]. Some of these compounds can be degraded by microorganisms in the activated sludge plant; however, a biorefractory portion of the effluent that

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Table 1 – Properties of the wastewater.

Property	WW1	WW2
Colour (PtCo)	182	23
pH	6.9	2.1
Total phosphorus (g m^3)	0.54	0.14
TOC (g m^3)	118	851

consist of high molecular weight oxidized lignin compounds are among those that are difficult to degrade [2,3]. There is also a concern about the load of phosphorus (P) and nitrogen (N) from the forest industry to water streams because of their contribution to eutrophication [4]. Most phosphorus in the wastewater naturally originates from the wood and their amount varies depending on the season [5].

A common first step to most biorefinery processes is a biomass pretreament to improve its subsequent availability to biomolecules or chemicals [6–8]. The pre-treatment typically consists either of an acid or an alkaline extraction usually performed under elevated temperature and strong shear (pressure or mechanical). For acid pretreatments, 1 kg t^{-1} to 2.5 kg t^{-1} of H_2SO_4 or SO_2 are typically used as catalyst combined with a steam explosion/extraction treatment. This step is expected to liberate the majority of the phosphorous, colour and organics in the process water effluents [9,10]. While significant information is available for effluents from alkaline extraction of ligno-cellulosics, thanks to the widely performed Kraft pulping process, much less is available for acidic treatments. Yet, acid catalyzed or autocatalyzed processes are becoming critical pretreatments for the biorefinery.

In this study, the water effluents of an integrated modern magnesium bisulphite pulp mill are investigated as a model for a biorefinery. The bisulphite process is an acidic pulping process that represents well an acidic pre-treatment. Multicomponent adsorption using lignite and activated carbon is studied as an economical tertiary process in complement to the usual water treatments. Colour, total organic carbon (TOC) and total phosphorus are the main concerns from water effluent of a would be ligno-cellulosic biorefinery [8].

In the first part of the study, the adsorption of model compounds on lignite and activated carbons is investigated using batch techniques. In the second part, effluents from a pulp and paper mill are treated by adsorption using activated carbon and lignite in batch and packed bed configurations. The properties of water (Total Organic Carbon (TOC), colour and total phosphorus) before and after treatment were compared, and the lignite selectivity to these contaminants is evaluated.

2. Materials and methods

2.1. Sample water

The wastewaters used in this study were the treated and untreated effluent from Kimberly Clark Australia (KCA) Tantanoola pulp and paper mill located at Snuggery in South East South Australia. The Tantanoola pulp mill produced 70 kt y^{-1} of magnefite chemical pulp and its water usage was about 60 m^3 t^{-1} of pulp. The logs were debarked before chipping. The feedstock consisted of *Pinus radiata* aged 12–15 years that was seasoned for 3 months to consume pitch, magnesium oxide (with less than 2.5% of calcium) and molten sulphur. The process was magnesium bisulphite at pH 4.5, temperature 170 °C and 6 h of batch cooking.

The effluent from the four tissue machines was combined to those of the pulp mill. The mill wastewater treatment plant consisted of two primary sedimentation clarifiers and three aerated stabilized basins in series for secondary treatment before discharge into a nearby lake [5].

WW1 identifies the clarifier effluent that had been subjected to aeration in a sedimentation pool that was collected at different times. WW2 identifies the evaporator clean acid distillate; this untreated effluent was the major contributor of organic contaminants of the wastewater. Basic water quality parameters that were determined for this water were TOC, pH, total phosphorus and colour, and these are provided in Table 1.

2.2. Preparation of model solutions

The lignite Loy Yang (LY) used in this study was from the Latrobe Valley mine, Victoria Australia and was used without any prior treatment. The lignite was sieved and the fraction having particles size in the range of 150 μm–212 μm was selected.

For comparison purposes, an activated carbon was also investigated. The activated carbon was a coal-based activated carbon purchased from Activated Carbon Technology Australia Pty., Eltham, Victoria. Proximate and ultimate analyses of the adsorbents are supplied in Table 2.

The model solution for colour adsorption was safranin (Basic Red 2, C.I. 50240), a cationic dye, purchased from Hopkin & Williams Ltd., Chadwell Heath England. The chemical structure of safranin is given in Fig. 1(a). Humic acid and KH_2PO_4, purchased from Sigma Aldrich Pty. Ltd., Sydney, Australia, were the model compounds for organic and phosphorus, respectively [11,12]. The exact chemical structure of humic acid is unknown: however several models of humic

Table 2 – Proximate and ultimate analyses of adsorbent.

	Proximate analysis (mass fraction %, db)			Ultimate analysis (mass fraction %, db)			Gross dry calorific value (MJ kg^{-1})
	Ash yield	Volatile matter	Fixed carbon	C	H	N	
Lignite	6.2	48.1	45.7	62.7	4.3	0.67	24.9
Activated carbon	12.6	2.1	85.3	83.3	0.5	0.4	28.4

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