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# Low-cost renewable adsorbent developed from waste textile fabric and its application to heavy metal adsorption

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ABSTRACT

Low-cost renewable adsorbent was developed from waste textile Lyocell via simple carboxymethylation and crosslinking reactions. The success of chemical modification was confirmed by scanning electron microscopy, energy disperse X-ray spectroscopy and Fourier transforms infra-red spectroscopy studies. Response surface methodology was employed to optimize the process parameters for higher performance. Factors such as sodium chloroacetate concentration and temperature in carboxymethylation and epichlorohydrin concentration for crosslinking, significantly affected the performance of the prepared adsorbent. Adsorption studies were conducted using Cd(II) as a model metal and the adsorbent was found to have ~17 times higher uptake than the original material. The results of this study may thus open a way to make useful heavy metal adsorbents from waste textile fibers to serve waste fiber recycling and heavy metal treatment purposes.

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## 1. Introduction

The use of low-cost waste materials to obtain value-added products is a desire of manufacturers and environmentalists as it adds up to a collective drive towards greener and cleaner environment [1]. This is particularly true when considering the development of adsorbents for treating metal-bearing water/wastewater. For a material to be considered for use as adsorbent, it must be cheap and go through safe processes (green routes) to arrive at the final product; it must be abundant in nature, and/or by-product from primary process [2,3]. Using low-cost waste material and adopting simple preparation method aid low pricing of adsorbents. Furthermore, the potential of regenerating and reusing adsorbents over several adsorption cycles is key to their large scale applications as this offers the advantage of reducing the cost of changing the adsorbents after every single cycle and also permits recovery of the adsorbates [4].

With the growing global population and the rising importance of textile polymers and nonwovens, the textile and fiber processing industries have formed part of the world largest production industries where majority of solid wastes are constantly generated [5]. Conventional solid disposal methods such as landfills and incineration are limited by strict environmental regulations, price escalations and greenhouse gases [6,7]. In constant efforts to reduce the growing effects of waste fibers which could not be curtailed by the traditional treatment methods, some recycling methods including reprocessing, depolymerization, composites, and heat regeneration, have been suggested and investigated [8-10]. However, despite some of these recycling methods proved to be good, their full exploits have been limited partly by process complexity, cost, technological, educational, and infrastructural inadequacy [7,11]. Meanwhile, an easier and cheaper recycling alternative could be suggested, and this is by using simple and eco-friendly chemical methods to convert these waste fibers into adsorbents to treat toxic pollutants. Cellulose-based materials, particularly in their modified forms, can display the required attributes of adsorbents, such as high uptake capacity, selectivity, regeneration and reusability [12-14].

In this study therefore, waste Lyocell fabric (LF) was used as a representative low-cost and waste material to develop heavy metal adsorbent via carboxymethylation reaction (CMR). CMR has been largely applied as a simple chemical process to prepare carboxymethyl cellulose (CMC) for applications in the pharmaceutical and food industries [15,16]. Response surface methodology (RSM) was employed in optimizing the reaction parameters for higher performance. The RSM is a collection of mathematical and statistical techniques used in building empirical models [17-19]. It provides effective platform for investigating factors and their interactions for desired response(s) in multi-factored experiment(s) [17,20]. In order to ensure cost-efficiency, the inexpensive and easy-to-handle reagents, i.e., sodium chloroacetate (SCA) and sodium hydroxide (NaOH) were used for the adsorbent

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**Nomenclature**

| | |
|---|---|
| CMR | carboxymethylation reaction |
| CMC | carboxymethyl cellulose |
| CLR | crosslinking reaction |
| RSM | response surface methodology |
| CCD | central composite design |
| ANOVA | analysis of variance |
| SCA | sodium chloroacetate |
| ECH | epichlorohydrin |
| LF | Lyocell fabric |
| SEM | scanning electron microscopy |
| EDX | energy disperse x-ray spectroscopy |
| FTIR | Fourier transforms infra-red spectroscopy |
| CMC-LF | carboxymethylated Lyocell fabric |
| CMC-LF-Cd | carboxymethylated Lyocell fabric after cadmium sorption |

manufacturing process. First, carboxymethyl cellulose (CMC) was produced by etherification of the –OH groups possessed by Lyocell with SCA in aqueous alkali [16]. Next, a crosslinking reaction using epichlorohydrin (ECH) enabled intralocking and interlocking processes between the remaining –OH groups and those of ECH, which provided water stability to the prepared adsorbent [15]. Factors considered in the RSM design include time, temperature and amounts of reagents used.

Due to the non-biodegradability and high toxicity associated with heavy metals, industries are always compelled to make every possible effort to control their discharge levels [3,21-23]. Cadmium is one of such toxic metals and is associated with many health issues such as renal dysfunction, hypertension, hepatic injury, and lung damage [21,23,24]. Cadmium and other heavy metals are reported to have caused serious contamination of soil and water bodies [25]. The presence of cadmium even in very low concentrations can endanger the ecosystem and so the World Health Organization has set a maximum guideline concentration of 0.003–0.005 mg/L in drinking water [25,26]. For the above reasons, the adsorbent prepared in this study was characterized and applied as a potential adsorbent to treat model cadmium-bearing wastewater. Detailed studies into the adsorbent preparation process and adsorption mechanisms were conducted and discussed accordingly.

## 2. Materials and methods

### 2.1. Materials

Isopropyl alcohol (99.5%), SCA (98%), and ECH (99%) were purchased from Sigma-Aldrich Co.; powdered NaOH from Daejung Chemical & Metals Co. Ltd; and methanol from SK Chemicals, Ulsan Korea. Ethyl alcohol (94%) and cadmium nitrate tetrahydrate were bought from Duksan Pure Chemical Co. and Junsei Chemical Co., respectively. All other reagents used were of analytical grades. LF was received as pre-consumer waste from fiber spinning and weaving company in South Korea.

### 2.2. Carboxymethylation reaction

The method of CMR was based on Williamson's ether synthesis [16,26], but with modifications to suit the current application. That is to a solution of isopropyl alcohol and water (80:20 by volume), 10 mL of 30% w/v NaOH was added and stirred, followed by 3 g of LF sample. The solution containing LF sample was pretreated in a shaking incubator maintained at 25 °C and 160 rpm for an hour, and 10 g of SCA was added to start the CMR. The temperature was then adjusted to 45 °C as the CMR proceeded for 3 h. The resulting carboxymethylated fibers were washed in methanol (~300 mL) to terminate the CMR.

### 2.3. Crosslinking reaction

After CMR, the carboxymethylated fibers became highly hydrophilic and soluble in water such that they could not be applied directly to heavy metal adsorption without crosslinking. Hence, crosslinking reaction (CLR) was necessary to provide stability to the adsorbent in solution. First, 5 g of NaOH was dissolved in 100 mL of ethyl alcohol in a glass bottle, followed by addition of 20 mL of ECH. The carboxymethylated fibers were then added, and the entire content was placed in a shaking incubator at 45 °C. After 24 h, the crosslinked fibers were taken out and washed thoroughly with methanol (~300 mL) and rinsed with distilled water (~2 L) until the pH was brought down to 7. The fibers were freeze dried to constant weight and labeled as CMC-LF thereafter.

### 2.4. SEM, EDX and FTIR characterization

The unmodified LF and CMC-LF together with CMC-LF after Cd(II) adsorption, were analyzed for confirmation of surface morphology augmentations and elemental peaks using a scanning electron microscope with energy disperse X-ray (EDX) functionality embedded (SEM, JEOL, JSM-6000 series WDS/EDS system, Japan). Fourier transforms infra-red (FTIR) spectroscopy analysis was performed with a Jasco FT/IR-4100 spectrophotometer to verify the introduction of new functional groups after chemical modification using the KBr disk technique. Small portions of the unmodified and modified samples were ground along with KBr and pressed under pressure for ~10 min to form transparent disks. The infra-red spectra of these transparent disks were then analyzed within the wavelength range of 4000 to 400 $cm^{-1}$.

### 2.5. Experimental design

After preliminary adsorption studies, performance of the prepared CMC-LF was found to highly depend on the amount (concentration) of SCA, CMR temperature, duration of CMR (time), and amount (concentration) of ECH used in crosslinking. Hence, the experiment was designed around these core variables, and their effects on the adsorbent's ability to effectively remove Cd(II), was the target response. The three factors of SCA concentration, CMR time, and CMR temperature, were varied in the CMR, and ECH concentration was varied in the CMR as the fourth factor. Central Composite Design (CCD), the most frequently used method in RSM design, was employed in this endeavor [17,20]. A four-factor ($n=4$) and five-level factorial design was set up within the experimental ranges chosen, based on the outcome of the preliminary studies. Coded and un-coded levels of the independent variables and the detailed CCD are respectively presented in Table S1 and S2 (Supporting Information). There were 27 experimental runs in all, with 16 experimental points, 8 additional points ($2n=8$) with axial distances of 2 ($\alpha=2n/4$), and 3 replicates at the central point of the design to evaluate and minimize experimental errors.

The individual factors and their interaction effects were simultaneously evaluated using a second-order polynomial model. In this model, the response variable (Cd(II) uptake) was expressed as a function of SCA concentration, $x_1$, CMR time, $x_2$, CMR temperature, $x_3$, and ECH concentration, $x_4$. The level values ($x_i$) of the independent variables were coded according to Eq. (1).

$$x_i = \frac{X_i - X_0}{\Delta X_i} \qquad i = 1,\ 2,\ 3,\ 4 \tag{1}$$

where $x_i$ is the dimensionless coded value of the $i$th independent variable, $X_i$; $X_0$ is the actual value of $x_i$ at the center point, and

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