

Research paper

The rationale for alternative fertilization: Equilibrium isotherm, kinetics and mass transfer analysis for urea-nitrogen adsorption from cow urine

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Received 21 September 2015; received in revised form 4 November 2015; accepted 5 November 2015

Available online 11 December 2015

Abstract

In an effort to minimize the loss of urea-N through volatilization and concurrently recover urea in a usable form from cow urine, adsorption experiments using bamboo-based activated carbon were performed. Batch studies were undertaken to evaluate the effect of initial concentration, sorption time and temperature on the variation in urea uptake capacity of the prepared carbon. Equilibrium data were tested against various isotherms, kinetics and mass transfer models. The Langmuir monolayer sorption was found to be 146.12 mg.g⁻¹ with nearly 90% urea recovery attained. The process was found to be reversible as seen through regeneration experiments. Thermodynamic parameters indicated that urea sorption was physical, spontaneous and exothermic in nature. Kinetic studies revealed that the rate of urea uptake was limited by both surface adsorption and intra-particle diffusion. It was concluded that the mass transfer of urea molecules over the activated carbon was governed by film diffusion at all concentrations examined.

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Keywords: Resource recovery; Waste recycling; Activated carbon; Wastewater reuse; Microwave; Sustainable agriculture

1. Introduction

Animal husbandry, and in particular, livestock wastes are a major source of ammonia (NH₃) in the atmosphere [1,2]. In recent years, the use of such nitrogenous sources in agriculture has increased, due to the ability of animal manure to supply plant-required nutrients to crops [3]. An impetus toward the use of these fertilizers is seen in organic farming where crop nutrients are exclusively supplied from such nitrogen (N) sources. Efficient manure management calls for the application of these wastes to arable land as a means for improving soil fertility and crop productivity. Cow urine is a rich source of nutrients; feedlot cattle is known to retain less than 20% of N from its diet resulting in more than 80% being lost in excretion [4]. Moreover, as Mobley et al. [5] point out, cow urine contains more than 90% of the excreted urea-N. While the use of urine as a

liquid fertilizer has been encouraged over the years, especially in contemporary organic farming settings [6], several studies have indicated that this approach could have considerable shortcomings. When added directly to soils, urine undergoes hydrolysis within a few days of application and volatilizes as ammonium-N [7]. Various investigations on NH₃ volatilization indicate that more than 50% of feedlot-N is lost to the atmosphere following the application of urine [8,9]. In addition, externalities in the form of eutrophication [10] and reduced soil N-fixing ability have also been recognized [11].

Although the rationale for recycling resources (plant-required) back to agricultural systems is justified for our resource-scarce society to continue to move toward its goal of ensuring food security, there is also an equally important need to address the shortcomings of our current efforts and look for alternative avenues. This investigation focuses on physical separation techniques (adsorption) as an effective strategy to recover urea-N from cow urine while addressing the limitations of previous research in this area [1–3,9]. Through our previous studies we have demonstrated that urea recovery could be realized using activated carbon [12,13]. This research examines the

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potential of bamboo shoots toward the recovery of urea-N from urine while simultaneously minimizing the loss of ammonium-N to the atmosphere. Specific emphasis has been extended to the equilibrium kinetics, isotherm and mass transfer analysis of the sorption to document the favorability of the investigated procedure vis-à-vis current alternatives. Urea-N recovery from cow urine through the procedure investigated here could offer insights into the discourse surrounding sustainable fertilization, resource recovery and recycling of organic wastes back to agricultural fields.

2. Methods and materials

2.1. Urine

Urine was collected from three Indian dairy cows (diet made up exclusively of grazed grass) in sterile polyethylene containers for a consecutive period of four days. The specimens were collected while the animals were indoor for milking. Following the collection all samples were immediately transferred to the laboratory and stored until further use at $-18\text{ }^{\circ}\text{C}$. Tests indicated that no loss of urea-N occurred during storage and refrigeration although *ca* 17% of the total N was observed to transform into ammonium-N when stored at room temperature ($23 \pm 2\text{ }^{\circ}\text{C}$). Kjeldahl analysis of urine allowed for determination of total N and urea-N to be 6.28 g.L^{-1} and 4.92 g.L^{-1} , respectively. Urea-N thus constituted 78.3% of the total N content in urine. Kool et al. [14] reported similar observations in their study. Hence, initial urea concentration in urine was analyzed to be 10.5 g.L^{-1} . Prior to the experimental runs all the samples were thawed, mixed uniformly and utilized immediately for sorption.

2.2. Activated carbon

Bamboo shoots procured from Lakimpur, Assam, India were initially washed with distilled water thoroughly to remove surface impurities, oven-dried for 24 h at $105\text{ }^{\circ}\text{C}$ and crushed in a roll mill to particle size of 10 mm. Following this, the shoots were microwave activated at 180 W for 10 min of irradiation time based on preliminary studies (CE104VD – Samsung, Malaysia). Subsequently, the activated precursors were placed in a porcelain boat and carbonized in a muffle furnace at heating rate of $24\text{ }^{\circ}\text{C.min}^{-1}$ and temperature of $500\text{ }^{\circ}\text{C}$ for 1 h. The carbon was allowed to cool in a desiccator to room temperature ($25 \pm 1\text{ }^{\circ}\text{C}$) and consequently size-reduced to 100 mesh (0.149 mm) in a ball mill (Deepthi Enterprise, Bangalore) at 80 rev.min^{-1} .

The activated carbon abbreviated as “BAC” throughout the manuscript was stored in air-tight polyethylene containers until further use. BAC was characterized for its surface and textural properties viz., surface area (BET): $911\text{ m}^2.\text{g}^{-1}$; pore volume: $0.2609\text{ cm}^3.\text{g}^{-1}$ and percentage moisture (ASTM D2867-09): 3.85. The iodine number (ASTM D4607-94) was determined using a 0.1 N standard iodine solution as $972\text{ mg of iodine/g of carbon}^{-1}$. The apparent density (ASTM D2854-09) was found to be 0.425 kg.m^{-3} , the porosity was 79.8%, average particle diameter $149\text{ }\mu\text{m}$, and total yield of the carbon was observed to be 28.5%. All reagents and chemicals utilized in the experi-

ments were purchased from Sigma Aldrich Chemicals, Mumbai, India and were of analytical grade.

2.3. Sorption experiments

Urine (50 mL) at various initial concentration (20% – 2.1 g.L^{-1} ; 40% – 4.2 g.L^{-1} , 60% – 6.3 g.L^{-1} ; 80% – 8.4 g.L^{-1} and 100% – 10.5 g.L^{-1}) was prepared through dilution with deionized water in Erlenmeyer flasks (250 mL). Sorption experiments were performed in an electric thermo-stated incubator shaker (Orbitek LT, Scigenics Biotech, India) at 150 rpm to determine the effect of initial concentration until the establishment of equilibrium by adding 3 g of BAC to 25 mL of the prepared urine solutions and studied at room temperature ($25 \pm 1\text{ }^{\circ}\text{C}$). Moreover, to understand the effect of temperature on the sorption, further experiments were performed at 30 and $35\text{ }^{\circ}\text{C}$ at various initial urea concentrations (20–100%) with constant adsorbent loading of 3 g and shaker speed of 150 rpm.

At various time intervals 1 mL aliquots of urine were withdrawn, filtered through a $0.45\text{ }\mu\text{m}$ syringe (Sartorius Stedim Boitech S.A., Germany) and monitored for change in absorbance (at 430 nm) using a UV-Vis Spectrophotometer (Shimadzu UV-1601, Japan). The procedure documented by With et al. [15] was followed to monitor the urea concentration at various time intervals. Sorption experiments were carried out in triplicate and the deviations were less than 5%. MATLAB® was used to perform all statistical analyses. In all graphical representations, average values have been reported. Eq. (1) was used to calculate the urea adsorption at equilibrium (q_e ; mg.g^{-1}) or urea uptake capacity [16]. C_0 and C_t (mg.L^{-1}) are urea concentration initially and at time ‘ t ’, respectively (min), V (mL) is the volume of urine used and W (g) is amount of BAC added.

$$q_e = \frac{(C_0 - C_t)V}{W} \quad (1)$$

Following the sorption, it becomes vital to subject the saturated carbon to regeneration for recovering the sorbate as well as to allow reutilization of BAC. Conventional regeneration of carbon is carried out through the application of high temperature thermal processes. However, these processes are unfeasible when it comes to recovery of volatile organics like urea. Chemical regeneration offers a promising approach but suffers from the risk of reaction of the reagent with the sorbate compounds and consequent generation of undesirable by-products thus necessitating post-regeneration separation. The present work thus investigates the efficacy of using deionized water as the desorption media for urea recovery. Regeneration experiments were performed by stirring 3 g of exhausted BAC in 150 mL of deionized water in a reciprocating shaker at $45\text{ }^{\circ}\text{C}$ and 150 rpm for 2 h (conditions were arrived at based on preliminary studies). Following this, the carbon was air dried at $105\text{ }^{\circ}\text{C}$ overnight and used for five additional sorption runs to evaluate its reusability potential over time.

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