Biodepuration of domestic sewage, textile effluents and acid mine drainage using starch-based xerogel from recycled potato peels

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ABSTRACT

Water is a finite resource. Its safety and cleanliness are highly important to meet current and future human needs. Compared to other resources, water represents a main factor to achieve development in several areas and leads to economic progress of a nation. However, in recent years, the excessive demographic and industrial growth has exacerbated water contamination. In this study, the biodepuration process of domestic sewage (DS), textile effluents (TE) and acid mine drainage (AMD) is conducted using starch-based xerogel from potato (Solanum tuberosum) peels. Results showed that the treatment is effective to achieve the reduction of 5-day biochemical oxygen demand. The most important result was the achievement of heavy metals removal for the three components. Firstly, there was a reduction of barium, zinc, and cadmium (91, 60 and 46%, respectively) for raw AMD. Secondly, there was a reduction in the levels of zinc, aluminum, and barium (89, 86 and 64%, respectively) for TE biodepuration. Finally, results showed a reduction in zinc, iron and cadmium levels (81, 78 and 57%, respectively) for DS biodepuration.

Key words | biodepuration, potato peels, starch, wastewater and xerogel

INTRODUCTION

One of the natural resources of great importance for the development of all economic activities and the survival of living beings is water. However, anthropogenic activities are generating serious impacts on the environment. For this reason, international development policies include environmental issues, social goals and promotion of the maintenance and care of this resource (Whittington et al. 2009).

Nowadays, water is being contaminated by the increase of domestic and industrial activities in our environment, generating detrimental changes in ecosystems and public health (Akar & Uysal 2010). The mining industry generates refractory compounds as water pollutants because of its extractive and refining operations. For instance, acid mine drainage (AMD) contains a high concentration of heavy metals, making it a potential contaminant of receptor bodies (Carrero et al. 2015). In addition, textile factories generate effluents, containing different kinds of synthetic dyes that even in minimum concentrations might cause environmental impacts in water bodies (Panic & Velickovic 2014; Sahinkaya et al. 2016). Finally, world demographic growth is causing an excessive increase in domestic sewage (DS) discharges in municipal and sanitary sewers. These discharges contaminate rivers and riverbeds when they are not properly treated (Ghorbel et al. 2017).

Different conventional electrocoagulation (Mamelkina et al. 2017) and non-conventional technologies (bioremediation and nanotechnology (Anjum et al. 2016; Ma et al. 2016)) for wastewater treatment are used with different levels of efficiency in many countries around the world. Nevertheless, it is important to develop new wastewater treatment technologies that can reduce energy and utilize agroindustry waste materials, such as the use of multiple types of modified starches from different origin, as it is reported in literature (Dahri et al. 2014; Rangabhashiyam & Selvaraju 2015).
Potato tubers (*Solanum tuberosum*) are the third most consumed food in the world, according to reports issued by specialized Peruvian organizations such as the International Potato Center. The worldwide industrial and domestic processing of potatoes generates peels and culls as residues in large quantities. They contain a wide variety of organic compounds such as starch, whose granules are in different integrity states, according to peeling method (Schieber & Saldaña 2009). Starch is the most abundant vegetal biodegradable polymer composed by amylose and amylopectin. These components confer potential properties for their physical and chemical modification with sewage depuration applications (Carvalho et al. 2003; Alvani et al. 2011).

Starch can be considered as a precursor of different semi-synthetic materials such as hydrogels (HG) that can remove and purify suspended and dissolved particles in water bodies, which are polluted by swelling and physical absorption (Güclü et al. 2009). Water-free HG is defined like xerogel. In addition, it has the same HG properties, and new features that confer the loss of water molecules (Kenar et al. 2014; Buscio et al. 2015).

The main objective of this study was to biodepurate three different kinds of residual effluents generated by the most common local anthropogenic activities, while using eco-friendly starch-based xerogel (XG) obtained from recycled potato peels, which are by-products of their industrial processing. In addition, physicochemical parameters and heavy metals were analyzed and quantified to determine the XG efficiency and biodepuration capacity.

**METHODOLOGY**

Potato peels used in this study belong to the OJO-AZUL variety (Peruvian native potato). These peels were residues of a local processing factory that uses potatoes to produce commercial products (frozen French fries and chips), and they are generally used as recycled animal feed (pigs, cows, rabbits, and others). This raw material was subjected to an aqueous homogenization process for the extraction of peel starch (PS) by using an electro-mechanical blender. Resulting suspensions were transferred to modified mechanical separators of Imhoff type in accordance with Coe and Clevenger (CC) settling theory for improving the static batch sedimentation process (Bürger & Wendland 2001). This modification consisted of the incorporation of a mechanical agitator in a hindered settling zone that inhibited the formation of the sedimentation critical point, allowing a greater amount of compacted starch to be produced. Finally, the starch was isolated from the rest of components by decantation, and it was subjected to two washing cycles using distilled water. Finally, it was stored and dried in a stove at 60 ± 5 °C for 48 hours to avoid physical and chemical damage.

The obtained PS sample was subjected to scanning electron microscopy (SEM) to determine its structural morphology. The device used was the scanning electron microscope VEGA II LSH. On the one hand, water absorption capacity (WAC) (g H₂O/g starch), solubility (SOL) (g soluble fraction/g starch) and swelling capacity (SC) (g gel/g soluble fraction) were determined at 80 °C (Crosbie 1991; Bryant & Hamaker 1997). On the other hand, dispersion clarity (DC), % Tₑ₅₀ was determined at 72 hours at 650 nm (Crosbie 1991) and retrogradation (R, %) at 4 °C after 7 days (Singh & Kaur 2004) in order to ensure the quality of the subsequently elaborated HG and XG. HG samples were prepared using a modification of the casting method reported by Pal et al. (2008), where 1,000 mL of a 2% starch solution was combined with 20 g of polyvinyl alcohol (PVA), to act as a plasticizer, with constant stirring until a homogeneous mixture was formed at room temperature. Crosslinking was achieved with the addition of 110 mL of alcoholic solution of glutaraldehyde (GA) and hydrochloric acid in isothermal and batch operation for 3 hours. This generated a pseudo-fluid mass known as an HG mixed with untransformed reagents. Subsequently, this sample was vigorously washed with distilled water to remove unreacted GA, PVA, ethanol and hydrochloric acid. Then, XG was obtained by drying it at standard temperature and pressure (STP) until a constant weight was generated. Finally, it was stored in hermetic bags for ensuring its conservation. XGs were characterized using SEM analysis and Fourier transform infrared spectroscopy (FTIR) with the transmittance method to characterize the presence of specific chemical groups in XG and verify their degree of crosslinking (Pal et al. 2008; Yu & Xiao 2008). Three different kinds of liquid effluents from different local anthropogenic activities were studied to determine XG biodepuration capacity. Grab samples were taken from a municipal sewer, textile factories and a typical Peruvian gold mine site located in the district of Ananea (San Antonio de Putina, Puno; 14°57′57″ S, 69°26′45″ W). Each of these effluents was physico chemically characterized by turbidity (TUR) (NTU), which was measured by using a portable VELB turbidimeter; electrical conductivity (EC) (µS/cm), which was measured with a Hanna HI-9628 conductivity meter; and dissolved oxygen (DO) (mg/L), which was...
measured with a portable Hanna meter HI-9146. These characteristics reveal changes occurring in biological parameters because of aerobic or anaerobic phenomena, providing data for the evaluation of the condition of streams of a river water (Chang 2005). Furthermore, chemical oxygen demand (COD) (mg/L), and 5-day biochemical oxygen demand (BOD₅) (mg/L) were determined.

Effluents discharged by domestic and industries into the surface and ground water contaminate the quality of the water, which can be assessed by BOD₅ determination (Sawyer et al. 1994). Heavy metals content was determined using inductively coupled plasma optical emission spectrometry (ICP-OES). For evaluating acid digestion, a pretreatment with a mixture of 4 mL concentrated nitric acid and 2 mL hydrogen peroxide was used for 5 minutes. After this time, digestion was performed, and the mixture was digested for 15 minutes. The first step was heating to 450 W for 5 min. The second step was holding at 650 W for 10 minutes. Lastly, cooling down was conducted at room temperature. The digested solution was diluted to 50 mL with ultrapure water and filtered using a cellulose membrane and stored at 5 °C until further analysis (Ahmed et al. 2017).

Each of the effluent samples obtained and characterized in the previous method was depurated with the contact of XG samples by using a jar test, which was constantly stirred at 650 rpm, where 500 mL of each effluent sample was contacted with 17 g of dry and powdered XG at room temperature for 48 hours. Finally, sedimentation of swollen XG was observed, which allowed recovery of the purified water that was later characterized and compared with original crude samples. All assays were repeated in triplicate.

Uptake of metal ions was calculated with the next equation:

\[ q = \frac{V \times (C_i - C_f)}{m} \]

where \( q \) is micrograms of heavy metal ions per gram of dry biosorbent; \( V \) is the reaction volume (L), \( C_i \) and \( C_f \) are the initial and residual metal concentrations (μg/L), respectively, and \( m \) represents the amount of dry biosorbent (g) (Tsekova et al. 2010).

**DISCUSSION AND RESULTS**

OJO-AZUL variety potato peels were selected and used as described in the methodology. Starch was extracted and isolated using Imhoff-type mechanical separators by applying CC sedimentation theory in order to obtain a higher efficiency of starch recovery. Therefore, these were called CC cones (Bürger & Wendland 2001). Starch samples were characterized by SEM analysis and high-resolution micrographs with an accelerating voltage of 20 KV (Figure 1). This revealed that starch granules were not contaminated with other compounds and preserved their own integrity. Size, shape and distribution of starch granules observed in the microscope at 136×, 272×, 545× and 1,089× magnifications indicate their potential use as precursors of HG. Moreover, it is also observed that granules showed a completely even and smooth surface. It proved they did not suffer any physical, mechanical or enzymatic damage or deterioration (Zhang & Oates 1999). The size, shape, and structure of extracted starch showed that it belonged to potato starch reported in literature. Size and shape of granules are directly related to the precursor biological source.

The size of the granules generally varies from 1 to 100 μm (Lindeboom et al. 2004). It was observed that there was a larger number of granules smaller than 20 μm at 136×, while granule abundance was recorded between 30 and 40 μm at 1,089×. Potato starch granules showed a round shape for the smallest, and an elongated spheroidal shape for the larger ones (Medina & Salas 2015). In addition, the analysis of micrographs at different magnifications showed regular edges and sizes grouped on the surface of potato starch obtained in accordance with the information reported by different authors in literature (Gunaratne 2002; Singh & Kaur 2004; Lizarazo et al. 2015).

Physicochemical characterization and functional properties of PS are shown in Table 1. They determined its quality and performance as a precursor of HG because of its significant influence on the synthesis of new structure. Values shown confirmed a high degree of crosslinking and extensive PS functionality as an ultimate precursor of XG (Crosbie 1991; Bryant & Hamaker 1997; Lovedeep et al. 2002).

Starch samples called PS had a lower WAC₈₀ value than ranges reported in literature (0.45–0.65 g/g; Nadir et al. 2015), probably due to the novel extraction method used in this study. Likewise, it was waste from a local potato processor. SC₈₀ value showed a lower value in potato starch samples compared to 52.81 g/g values reported by Hoover in 2001 cited by Nadir et al. (2015). SOL₈₀ value indicated that the soluble fraction was greater than 90% (0.92 g/g) in contrast to the values reported by the same authors (12–14 g/g), according to DC₇₂ value obtained in laboratory assays. For R₇ value, it resulted in a key feature of functionality of the recovered starch because high values indicated the disintegration of amyllose and amylopectin networks to
form new modified structures confirmed by FTIR spectra, since OJO-AZUL PS had abundant hydroxyl groups, which could be used to easily prepare HG (Tako et al. 2014).

OJO-AZUL PS obtained and characterized was considered as an HG precursor because of the properties shown in Table 1 and previously discussed. Figure 2(a) shows a digital photograph of the macroscopic morphology of starch-based HG, which shows a high thickness, non-regular, corrugated, smooth consistency, high humidity, homogeneous white color, and tensile strength surface (Sid-daramaiah et al. 2005). These properties were obtained by the addition of reagents such as PVA, GA, ethanol, and hydrochloric acid, as indicated in detail in the ‘Methodology’ section. On the other hand, the reaction of PVA with GA was normally carried out as reported in international literature, forming acetal bridges among the pendant hydroxyl groups of PVA chains, which is known as crosslinking. This reaction was potentiated using hydrochloric acid as a catalyst (Mansur et al. 2014). The final XG was obtained from uniform drying of HG at STP (Kenar et al. 2014), showing new macroscopic characteristics like smaller thickness than its predecessor, flat and smooth surface, besides a remarkable gravimetric difference that resulted in the near total loss of free water. It also showed a semi-translucent whitish color, and a solid and resistant consistency, as shown in Figure 2(b). The main physical and even organoleptic difference between both modified structures of the precursor starch is focused on their water content.

Furthermore, Figure 3(a) shows a high-resolution electron micrograph of the XG surface in which remnants of apparently intact agglomerated starch granules with notable surface changes are observed. These are not smooth or uniform like the surface of PS shown in Figure 1. The irregular surface has well demarcated elevations and depressions, and a solid matrix, enveloping the highly porous starch granules, which give it desirable properties for swelling and water

| Functional properties of OJO-AZUL PS |     |     |
|-------------------------------------|-----|-----|
| Physicochemical property            | Value | Unit       |
| WAC<sub>80</sub>                   | 0.1969 ± 0.95 | g H₂O/g starch |
| SOL<sub>80</sub>                   | 0.92 ± 0.72   | g soluble fraction/g starch |
| SC<sub>80</sub>                    | 14.51 ± 1.48  | g/g soluble fraction |
| DC<sub>72</sub>                    | 3.82 ± 0.23   | % T₆₅₀ |
| R₇                                 | 64.50 ± 3.61  | % |
absorption. These are observed in the dark fraction of Figure 3(b) and have porosity with an average size of approximately 2 μm (Glenn et al. 2011).

FTIR characterization of the XG structure showed in its spectrogram the following 20 sequential transmittance peaks (% T) at 759.9, 844.86, 925.87, 1003.05, 1014.60, 1080.18, 1145.77, 1207.49, 1238.35, 1334.80, 1373.38, 1419.67, 1450.53, 1643.42, 1743.72, 1979.05, 2164.22, 2360.97, 2920.35 and 3317.71 cm$^{-1}$.

The band between 3,200 and 4,000 cm$^{-1}$ represented the O-H section. The absorption peak at 2,900 cm$^{-1}$ represented the C-H stretch. There was no absorption of aromatic rings in the typical region (1,500–1,600 cm$^{-1}$). However, the intense absorption at 1,643 cm$^{-1}$ would be a result of water molecules in amorphous regions of the

Figure 4 shows up to seven sections of absorption bands for O-H, C-H, C-O-H, C-O-C, C-O, C-C and pyranose-glucose rings (Kemas et al. 2012).

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biomolecule of the starch analyzed. The absorption peak at 1,145 cm\(^{-1}\) represents C-C coupled mode and vibrations in the C-O stretch, while the absorption peak at 1,080 cm\(^{-1}\) represents the band with a possible flexural vibration in C-O-H bonds. The absorption at 925 cm\(^{-1}\) is typical for the C-O-C system in basic vibration mode of the \(\alpha\)-1,4-glycosidic bond, whereas 589 and 461 cm\(^{-1}\) represent the basic mode of the pyranose ring, which was not obtained in this spectrum such as described in literature. In addition, the FTIR spectrum for XG is comparable with a high degree of significance with the FTIR spectrum for the potato starch shown in Figure 5 (Dupuy et al. 1997; Dolmatova et al. 1998; Cerna et al. 2003; Kemas et al. 2012).

Figure 5 shows the comparison of FTIR spectra of PS and XG, with great similarity between the spectrograms even up to 91–93% probability. The starch spectrum was obtained from the 901 Nicodom IR Suspect Powders Spec Library (Nicodom Ltd).

HG has a crosslinked structure that is conducive to the development of biosorption. This mechanism involves a solid phase (sorbent) and a liquid phase (solvent) that species contain to be adsorbed. There are complexation and adsorption mechanisms in the superficial pores. There is a process of ion exchange or adsorption by physical forces in the spaces of the structural network and in inter- and intrafibrillar capillaries. Characteristics mentioned above provide very interesting properties to the material to remediate contaminated water (Zheng et al. 2010; Muya et al. 2015).

DS treated with XG showed a significant TUR and EC reduction of 47.4% and 87.5%, respectively (Table 2). TUR and EC reduction are closely related to soluble and suspended solids removal, which increased oxygen solubilization capacity in depurated water bodies (Mitchell et al. 1999). In this study, it was verified that DO concentration increased in all cases with the reduction of the pollutant content of depurated water. Furthermore, pH values do not show a significant variation, which ensures the neutrality of XG used. Percentage removal was 80.4% in the case of BOD\(_5\), and 34.5% for COD. Results demonstrated the interesting properties of removing a higher fraction of organic matter compared to inorganic matter.

Textile effluents (TE) show a reduction in TUR and EC of 60.7% and 39.0%, respectively (Table 3). It can be observed that pH value does not have a significant variation.
Removal percentage was 88.2% in the case of BOD$_5$, and 20.2% for COD.

AMD shows a reduction in TUR and EC of 33.3% and 36.0%, respectively (Table 4). It can be seen that pH value does not have a significant variation. Removal percentage was 63.7% in the case of BOD$_5$, and 28.4% for COD.

Tables 5–7 show removal percentage and adsorption capacity of Al, Ba, Cd, Fe, Ni, Pb and Zn in samples of

![Figure 5](image-url)  
**Figure 5** | FTIR spectrum of XG (top) and potato starch (bottom) provided by a spectral library (Nicodom Ltd).

| Parameters | Raw | Depurated | Legislation$^a$ | Compliance$^b$
|---|---|---|---|---
| TUR (NTU) | 62.70 ± 0.51 | 29.73 ± 0.25 | n.s.$^c$ | n.s.
| EC (μS/cm) | 1,120 ± 2.41 | 980 ± 2.56 | n.s. | n.s.
| DO (mg/L) | 1.02 ± 0.09 | 3.57 ± 0.21 | n.s. | n.s.
| pH (-) | 6.99 ± 0.14 | 7.14 ± 0.20 | 6.5–8.5 | ✓
| BOD$_5$ (mg/L) | 2,700 ± 3.06 | 530 ± 1.53 | 100 | ✗
| COD (mg/L) | 3385.1 ± 2.67 | 2218.5 ± 2.54 | 500 | ✗

$^a$Peruvian Legislation 2017.
$^b$Compliance under Peruvian legislation.
$^c$Not specified.
DS, TE and AMD, analyzed in triplicate by ICP-OES equipment with sensitivity, precision and accuracy. Table 5 shows that the most outstanding removal percentages for DS were for Pb, Fe and Ni with values of 80.67, 78.35 and 56.67%, respectively. Additionally, adsorption capacity is considerable for Fe and Pb with 205 and 8.89 μg/g, respectively.

HG and XG had properties such as having a lighter hydrophilic structure than water, the suitability of the monomeric functional groups for direct synthesis, and additional penetration into the contaminants due to their three-dimensional structures. In addition, the research reports the removal of heavy metals (Zn and Ni) from synthetic industrial effluents (Sezgin & Balkaya 2018). XGs are composed of starch in this study. Starch is a polymer that contains hydroxyl and carboxyl groups. These are interlinked with metal ions to form metal complexes (Ciesielski & Tomasik 2017; Ciesielski & Krystyjan 2013; Al-Qahtani 2019).

Table 6 shows that, for TE, the most significant percentages are Pb, Cd, Zn and Fe with a removal percentage of 89.25, 85.71, 64.52 and 60.63%, respectively. Concentration of heavy metals is the lowest of the three effluents used in the study. Therefore, the adsorption capacity is very low.

Results obtained for the removal of Fe in DS and TE show an efficiency of 78.35 and 60.63%, respectively, in Tables 5 and 6. These results are similar to those obtained by other researchers (El-Hag Ali et al. 2003; Yetimoglu et al. 2007; Abd El-Mohdy et al. 2015; Huang et al. 2016).
Results show Zn removal efficiency in TE and AMD of 45.71% and 91.02%, respectively. Likewise, the table shows considerable adsorption capacity of Fe, Al and Ba with 64.52, 91.02 and 60.29%, respectively. These results are similar to those reported by Souda & Sreejith (2015). However, efficiency is not good for Pb removal in AMD. A removal efficiency of 85.71% Cd can be observed in TE in presence of heavy metals (Al, Ba, Cd, Fe and Zn) determined a removal percentage in all cases. In addition, the depuration of these contaminants in any receiving body.

The XG obtained from husks and debris of local industries of potato processing demonstrated partial biodepuration capacity for three different types of wastewater from anthropogenic activities that are common in the local area (DS, TE, and AMD). Results showed a significant reduction in BOD₅, TUR, COD, and EC, according to an increase in DO concentration in all cases. In addition, the depuration of heavy metals (Al, Ba, Cd, Fe and Zn) determined a removal percentage in accordance with the following order: Ba > Zn > Al > Fe > Cd, considering the three types of wastewater treated with XG. The XG was able to improve water quality by removing these metals at certain concentrations that are detrimental to human health and ecosystems. In conclusion, it is proposed that XG can be an effective agent for the final treatment of DS, as it has high levels of BOD₅ reduction, and especially can be used for removal of heavy metals such as Ba, Zn, Al, Fe and Cd in wastewater produced by different anthropogenic activities.

**CONCLUSIONS**

The XG obtained from husks and debris of local industries of potato processing demonstrated partial biodepuration capacity for three different types of wastewater from anthropogenic activities that are common in the local area (DS, TE, and AMD). Results showed a significant reduction in BOD₅, TUR, COD, and EC, according to an increase in DO concentration in all cases. In addition, the depuration of heavy metals (Al, Ba, Cd, Fe and Zn) determined a removal percentage in accordance with the following order: Ba > Zn > Al > Fe > Cd, considering the three types of wastewater treated with XG. The XG was able to improve water quality by removing these metals at certain concentrations that are detrimental to human health and ecosystems. In conclusion, it is proposed that XG can be an effective agent for the final treatment of DS, as it has high levels of BOD₅ reduction, and especially can be used for removal of heavy metals such as Ba, Zn, Al, Fe and Cd in wastewater produced by different anthropogenic activities.

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