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Preparation of chitin/PVA/SF ternary blend for heavy metal ion removal from electroplating industrial effluent

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ABSTRACT

The tremendous increase in the use of heavy metals over the past few decades has inevitably resulted in an increased flux of metallic substances in aquatic environment. Mine drainage, industrial and domestic effluents, agricultural run-off, acid rain etc. have all contributed to some extent to the metal loads in the water bodies. Metals are of special concern because they are non-degradable and therefore persistent. Some metals may be either beneficial or toxic, depending on concentration. The efficient removal of toxic metals from wastewater is an important matter and it is a widely-studied area. There are many techniques available for the removal of heavy metals such as chemical precipitation, reverse osmosis, electrolysis and ion exchange. Physical treatment can also be used to remove small concentrations of hazardous substances dissolved in water that would never settle out. One of the most commonly used techniques involves the process of adsorption, which is the physical adhesion of chemicals onto the surface of a solid. The effectiveness of the adsorbent is directly related to the amount of surface area available to attract the molecules or particles of contaminant. As the use of activated carbon is expensive, so there has been considerable interest in the use of other sorbent materials. Among the many other low cost absorbents identified chitin has the highest sorption capacity for several metal ions but has some mass transfer problems. In the present investigation an attempt was made to overcome these mass transfer limitations by synthesizing binary blend using chitin and poly vinyl alcohol; and ternary blend using chitin, poly vinyl alcohol and silk fibroin and the both blends were characterized by FTIR, TGA, XRD and SEM. From the characterization results, it was found that chitin/PVA/SF (1:1:1) ternary blend is thermally stable than chitin/PVA (1:1) binary blend. Thus, electroplating industrial wastewater was treated with the prepared ternary blend and results revealed that the ternary blend prepared was excellent in removing the heavy metal ions from electroplating industrial effluent. Hence, the ternary blend of chitin, poly vinyl alcohol and silk fibroin could open way for waste water treatment in industrial level.

Introduction

Human activities are intimately tied to the environment, and harmful behaviors often have harmful consequences. In order to ensure healthful environment for the future of humanity it is necessary to reduce the harm done to the environment through contamination. Amongst the countless man-made contaminants that infiltrate our water sources are heavy metals. Heavy metal contamination of water resource is of great concern because of the toxic effect to human beings and other animals and plants in the environment at even very low concentrations (Kadirvelu et al., 2000; Descalzo et al., 2003; Xiao and Thomas, 2005). Heavy metal solutions have been widely used in electroplating, metal finishing, dying surface treatment industries, etc. It is well known that heavy metals such as Cu, Co, Ni, Cd, and Zn are usually associated with tendency to accumulate in living organisms, and are highly toxic when adsorbed into body. These are serious threat to human populations as well as the fauna and flora of receiving water bodies as discharged in waste water (Dantas et al., 2001). Since heavy metal ions are not biodegradable in nature, effective removal of heavy metal ions through physical or chemical technologies is important in the protection of environment quality and public health.

In order to minimize the adverse effects of toxic metal in the environment, there are various methods for the removal of toxic metals from aqueous solutions such as chemical precipitation, evaporation, ion exchange, adsorption, cementation, electrolysis and reverse osmosis (Crist et al., 1996) usually called conventional adsorption. These processes may be ineffective or expensive (Volesky and Holans, 1995) especially when the heavy metal ions are in solutions containing in the order of 1- 100 mg dissolved heavy metal ions/L (Volesky, 1990a,b). As a result of these, biological methods such as biosorption/bioaccumulation for the removal of heavy metal ions may provide an attractive alternative to physico-chemical methods (Kapoor and Viraraghavan, 1995; Pagnanelli et al., 2000).

Biosorbents are prepared from naturally abundant and/or waste biomass. Due to the high uptake capacity and very cost-effective source of the raw material, biosorption is a progression towards a perspective method. Recently numerous approaches have been studied for the development of cheaper and more

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effective adsorbents containing natural polymers. Many biosorbents such as fungi (Acosta et al., 2004), algae (Gupta et al., 2001), seaweeds (Kratochvil et al., 1998; Elangovan et al., 2008), microorganisms (Sahin and Ozturk, 2005; Fan et al., 2008), and several biopolymers (Wu et al., 2008; Bailey et al., 1999) have been utilized in the removal of heavy metals from wastewater. The polysaccharides are renewable resources which are currently being explored intensively for their applications in water treatment (Gupta and Ravikumar, 2000). Among various polysaccharide compounds, chitin and their derivatives (Ravikumar, 2000), deserved particular attention. These polysaccharides are abundant, renewable, and biodegradable, low – cost and are the best choice in water treatment and useful tool for protecting the environment (Bolto, 1995). Chitin, a polymer composed of n-acetyl D-glucosamine residue extracted from crab and shrimp shells, is the second most abundant resource (next to cellulose) in nature. It is worth noting that both chitin and chitosan are recogonized as excellent metal ligands, forming stable complexes with many metal ions (Chui et al., 1996). Chitin is superior to chitosan, especially in the biomedical fields, due to the fact that acetamide group present in chitin is similar to the amide linkage of protein in living tissues (Muzzareli, 1985) making chitin more bio compatible than chitosan.

Blending is an especially important process for developing industrial applications of polymeric materials and compatibility among components has a marked influence on the resulting physical properties of polymer blends (Folkes and Hope, 1985). Blending a natural polymer with a synthetic one seems to be an alternative way of preparing polymeric alloys to meet specific applications. Poly(vinyl alcohol) (PVA) is a non toxic, water soluble, synthetic polymer that is widely used in biomedical applications with its excellent film-forming ability. PVA is a good candidate for use as membranes and hydrogels (Hassan and Peppas, 2000; Kim et al., 1992). Mechanical properties of chitin can be improved through blending with poly(vinyl alcohol) which is highly elastic in spite of its crystalline feature and is also biocompatible material.

Silk fibroin (SF) is a natural fibrous protein spun from *Bombyx mori* silkworm and is composed of a repetitive sequence of amino acids: glycine, alanine and serine, and as all fibrous proteins is not soluble in water due to its high concentration of hydrophobic amino acids (Altman et al., 2003). SF films become brittle in the dry state and would be unsuitable for practical use (Li et al., 2002). If the dry state is required and the brittleness is undesirable, SF properties can be improved by blending with other natural biopolymers (Kweon et al., 2001; Lee et al., 2004; Vasconcelos et al., 2008; Marsano et al., 2007).

In present work, we attempted to prepare binary and ternary blend of chitin (CT) with PVA and SF. The films formed were characterized by FTIR, TGA, SEM and XRD. FTIR spectra and SEM analysis showed proper blending has taken place between the polymers. From the characterization results, it was found that chitin/PVA/SF (1:1:1) ternary blend is more stable than chitin/PVA (1:1) binary blend. Thus, the synthesized ternary blend (1:1:1) was subjected to electroplating industrial effluent in order to determine the extent of adsorption. The extent of removal of the heavy metals was investigated by changing the adsorbent dose, pH of the solution and contact time and the result revealed that the synthesized blend film was excellent in removing the heavy metals ions.

Methods and materials

Materials

Chitin was received from India seafoods, Cochin. Cocoons of *Bombyx mori* were obtained from the sericulture farm in Vaniyambadi, Vellore District. Poly vinyl alcohol and other reagents were of analytical grade and used as received. Effluent from the electroplating industry, SIPCOT, cuddalore, Tamil nadu, India was collected.

Preparation of blends

The chitin, polyvinyl alcohol binary blend was prepared by mixing solutions of chitin with polyvinyl alcohol solution in the weight ratio 1:1 and 10 ml formaldehyde was added as cross linking agent to the mixture. The solution was stirred well and was stored at 5 °C overnight and then allowed to dry to get chitin/polyvinyl alcohol blend. Similarly, chitin, polyvinyl alcohol and silk fibroin ternary blends were prepared by mixing solutions of chitin with polyvinyl alcohol and silk fibroin solution and the weight ratio 1:1:1 and 10 ml formaldehyde was added as cross linking agent to the mixture. The solution was stirred well and was stored at 5 °C overnight and then allowed to dry to get chitin/polyvinyl alcohol/silk fibroin ternary blend.

Characterization

The prepared blend films were analysed by FTIR in a wide range wavelength between 400 cm^{-1} and 4000 cm^{-1}, and in solid state using KBr pelletisation. A Perkin – Elmer spectrophotometer was used. Thermo gravimetric analysis was carried out in nitrogen atmosphere at a heating rate of 15 °C/min up to temperature range of 1400 °C on NETZSCH-STA 409c/CD thermal analyzer. SEM study of the prepared blend films were carried out by JSM – 640 Scanning Electron Microscope, JEOL at 20MA and 15KV. The dried sample film was cut and was sputter – coated with gold using a microscope sputter coater and viewed through the microscope. X-ray diffraction studies were performed using X-ray powder diffractometer (XRD – SHIMADZU XD – D1) using a Ni-filtered Cu Kα X-ray radiation.

Physiochemical characterization of Effluent

Physicochemical factors such as pH, electrical conductivity (EC), dissolved oxygen (DO), biochemical oxygen demand (BOD), chemical oxygen demand (COD), total dissolved solids (TDS), total suspended solids (TSS), total solids (TS), alkalinity, chloride, hardness, sodium and heavy metals such as cadmium, copper, cobalt, nickel, chromium, lead and zinc were analyzed as per the methods (Eaton and Franson, 2005; Trivedy and Goel, 1984).

Results and Discussion

Fourier transform infrared spectroscopy (FTIR)

As shown in Fig. 1(a), the spectrum of pure chitin film shows a broad band at 3434 cm^{-1} which is due to the OH stretching. The band at 1561 cm^{-1} is assigned for the NH bending (amide II) (NH_2) while the small peak at 1654 cm^{-1} is attributed to the C=O stretching (amide I) O=C-$NHCH_3$. The bands at 2926, 1414, 1317 and 1262 cm^{-1} are assigned to CH_2 bending due to pyranose ring. The band at 1378 cm^{-1} is due to CH_3 wagging. Figs. 1(b) and (c) present the FTIR spectrum of the chitin/PVA (1:1) binary film and chitin/PVA/SF (1:1:1) ternary blend film. As can be seen, the presence of PVA in the chitin caused remarkable shift for the C=O stretching peak at 1654 cm^{-1} of chitin to a lower wave number at 1650 cm^{-1}, whereas in chitin/PVA/SF (1:1:1) ternary blend, the presence of SF in the binary blend film caused significant decrease in the wave number up to 1629 cm^{-1}. In addition, the bands at 2926

cm^{-1} and 1317 cm^{-1} of chitin disappeared in the spectra of the binary blend as well as in ternary blend. This is due to the fact that when two or more polymers are mixed, changes in characteristic spectra peaks occur due to the reflection of the physical blends and chemical interactions. These observations indicate the existence of good miscibility between chitin and PVA and SF and this is most likely due to the formation of intermolecular hydrogen bonds between the amino and hydroxyl groups in chitin and in SF and the hydroxyl groups in PVA.

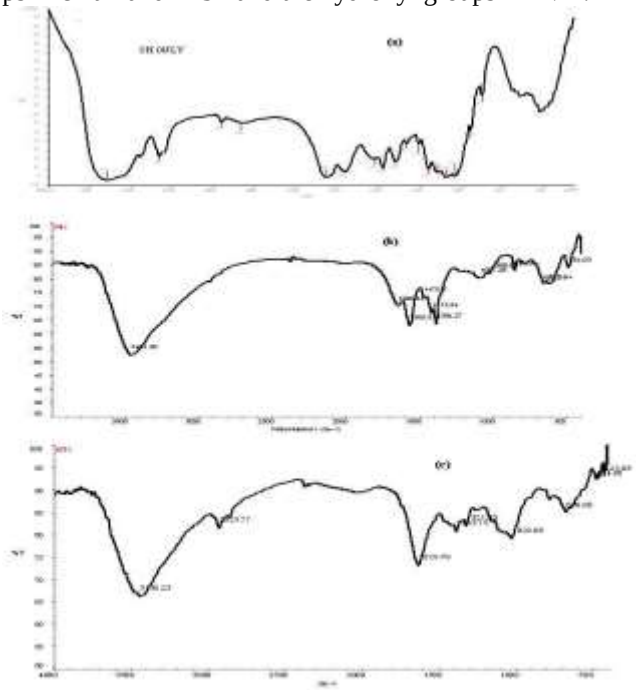

Fig. 1. FTIR spectra of chitin (a), chitin/PVA binary blend (b) and chitin/PVA/SF ternary blend (c).

X-ray diffraction

X ray diffraction is a proven tool to study crystal lattice arrangements and yields very useful information on degree of sample crystallanity. X-ray pattern of chitin, chitin/PVA (1:1) binary blend, chitin/PVA/SF (1:1:1) ternary blend are shown in Fig. 2. Pure chitin shows sharp and narrow peak at 2θ =9.32° and 19.25° which are associated with the crystalline region.

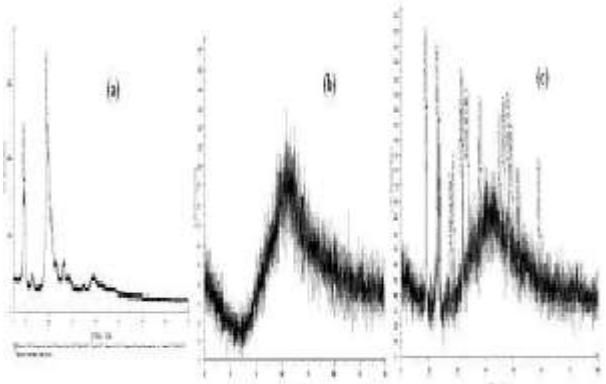

Fig. 2. XRD patterns of (a) chitin, (b) chitin/PVA binary blend and (c) chitin/PVA/SF ternary blend

In chitin/PVA (1:1) binary blend, the peak of the hydrated crystalline structure of chitin at 9.32° is absent and the strong 19.25° peak is diminished. There is broad peak in the region between 40°and 44°, which may have contribution from both the chitin and polyvinylalcohol. It illustrates that existence of PVA decreases the crystallinity of chitin in the binary blend. In chitin/PVA/SF (1:1:1) ternary blend, XRD pattern shows two sharp peaks at 18.86 and 23.04° and a broad peak in the region between 41° and 43°. This result shows that the presence of silk fibroin induced several crystalline forms. However, the existence of SF decreases the crystallinity of the ternary blend. This phenomenon is due to the significant hydrogen bonding interaction among CS, PVA, and SF molecules. In other word, the addition of SF improves the compatibility between CS and PVA.

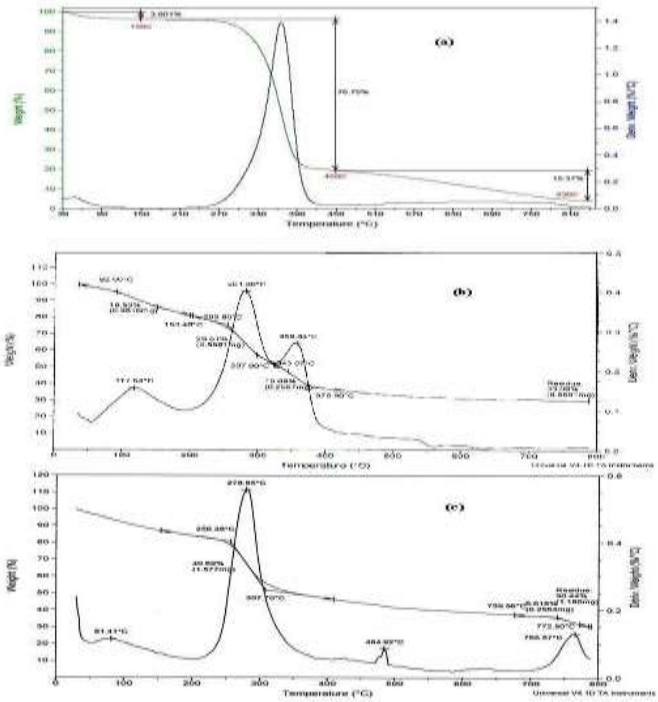

Fig. 3. TG curves of (a) chitin, (b) chitin/PVA binary blend and (c) chitin/PVA/SF ternary blend.

Thermo gravimetric analysis

TGA is a useful technique to assess the thermal stability of polymer and polymer blends. Decomposition and thermal stability of chitin, and blended films were determined from thermograms as shown in Fig. 3. Fig. 3(a) shows the thermogram of pure chitin. Two weight losses are observed in the chitin TGA curve. The weight loss around 150 °C is due to moisture vaporization. The other weight loss at 270-390 °C is due to the degradation of chitin molecules. In the case of chitin/PVA (1:1) binary blend, three weight losses due to evaporation of water, breaking of chitin/PVA linkages and degradation of chitin and PVA into smaller components at 117.54 °C, 281.36 °C and 358.44 °C, respectively, whereas, chitin/PVA/SF (1:1:1) ternary blend exhibits four weight loss steps due to the evaporation of water, breaking of chitin/PVA/SF linkages, degradation of chitin and the degradation of individual polymer into smaller components at 81.41 °C, 278.95 °C, 484.92 °C and 765.57 °C, respectively. From the thermogram, it is evident that at 772 °C, 95% of chitin, 71.69% of chitin/PVA binary blend and 69.56% of chitin/PVA/SF ternary blend undergo degradation. The residue after degradation for chitin 5%, for chitin/PVA 28.31% and for chitin/PVA/SF 30.44% was observed. From the results, it was concluded that the thermal stability is in the order chitin/PVA/SF > chitin/PVA > native chitin.

Morphology

The scanning electron micrograph of chitin/PVA binary (1:1), chitin/PVA/SF ternary blend (1:1:1) are shown in Fig. 4.

The blending of PVA with chitin modified the surface morphology of chitin. SEM image of chitin (not shown) was smooth, homogeneous and continuous matrix without any pores (or) semi-pores (or) cracks on the surface with good structural integrity. The SEM image of chitin/PVA revealed the uniform distribution of PVA in chitin matrix. The SEM image of ternary blend of chitin/PVA/SF showed that the blending has improved porosity and fractured structure, which can be responsible for adsorption of molecules. The surface area of chitin/PVA/ternary blend films is highly rough in comparison with binary blend film and chitin. This obviously accounts that ternary blend has good adsorbing property which is useful for wastewater treatment.

Adsorption Studies

From the above characterization results, it was found that chitin/PVA/SF (1:1:1) ternary blend is more stable than chitin/PVA (1:1) binary blend. Thus, electroplating industrial wastewater was treated with the prepared ternary blend.

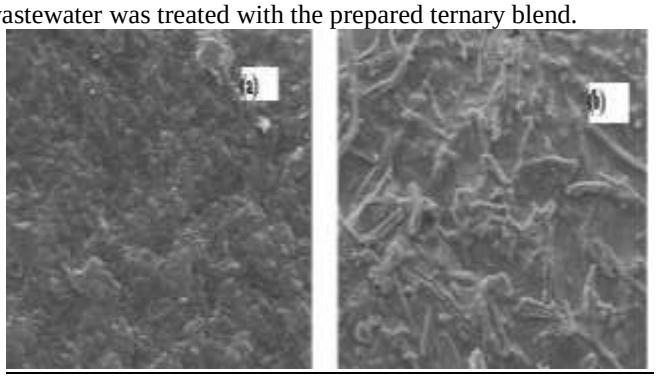

Fig. 4. SEM images of (a) chitin/PVA binary blend and (c) chitin/PVA/SF ternary blend.

Effect of adsorbent dosage in electroplating industrial effluent

Table 1 represents the physico chemical parameters and heavy metal content of the electroplating industrial effluent collected from an industry SIPCOT, Cuddalore, Tamilnadu, India

All the parameters along with the heavy metal contents were found to be very high than the accepted limits.

Various dosage of chitin/PVA/SF(1:1:1) blend have been used to treat electroplating industrial effluent. The parameters such as BOD, COD and ions such as chlorides, sodium, magnesium and also heavy metals such as copper, nickel, chromium, cadmium, zinc and lead have been drastically decreased with the increase in adsorbent dosage. The adsorption of heavy metal ion is maximum at 4 g/L which is a minimum amount. Hence 4 g/L, was concluded as the optimum dosage of the ternary blend (CT/PVA/SF 1:1:1) in treating the effluent.

Effect of contact time in electroplating effluent treatment

Table 2 represents the effect of contact time on the treatment of electroplating industrial effluent by the ternary blend [CT/PVA/SF 1:1:1]. On increasing the time, the initial effluent concentration gradually reduced, after 3hours all the parameters like [COD,TS] reduced to maximum extent and attained equilibrium. Hence 3hrs was concluded as an optimum contact time**.**

Effect of pH on electroplating effluent treatment

Table 3 represents the effect of pH on electroplating industrial effluent treatment. The reduction of all the parameters by the treatment of ternary blend was found to be pH dependent. From the results it is evident that there was a maximum adsorption of all the parameters by the ternary blend at pH 5 and hence pH 5 was found to be the optimum pH for treating electroplating industrial effluent.

Conclusions

Our research demonstrate how a new type of adsorbent, chitin/PVA/SF (1:1:1) ternary blend, can be synthesized through solution blending for the removal of heavy metal ions from the electroplating industrial effluents. The adsorption capacities of the ternary blend were dependent on the dosage of adsorbent, pH and the contact time. In addition, the study concluded that the use of chitin/PVA/SF (1:1:1) ternary blend for heavy metals removal appears to be technically feasible, eco-friendly and with high efficacy. We hope that our chitin/PVA/SF ternary blend adsorbent will find more practical use in the near future.

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Table 1. Effect of adsorbent dose on the treatment capacity of CS/PVA/SF(1:1:1) of electroplating industry effluent

Parameters	Raw effluent	Adsorbent dose			
		1 g/L	2 g/L	3 g/L	4 g/L
EC (μmhos)	13215	1225	718	559	425
TSS (mg/L)	387	225	145	105	101
TDS (mg/L)	15556	8125	510	410	400
BOD (mg/L)	8865	1150	741	345	310
COD (mg/L)	3125	4250	2110	1210	1110
Chlorides (mg/L)	15111	4515	1110	280	210
Sodium (mg/L)	2440	1121	750	410	410
Potassium (mg/L)	1896	810	400	100	89
Magnesium (mg/L)	520	205	177	75	72
Calcium (mg/L)	340	210	112	25	20
Zinc (mg/L)	35	15	9	6	5
Chromium (mg/L)	112	25.1	15	8.5	7
Nickel (mg/L)	46	15	10	8	5
Copper (mg/L)	55.5	28.5	20	14	6
Cadmium (mg/L)	7.8	5.1	3.0	2.1	2.0
Lead (mg/L)	7.5	4.1	3.4	2.2	2.0

Table. 2 Effect of contact time on the treatment capacity of CS/PVA/SF (1:1:1) of electroplating industry effluent

Parameters	Raw effluent	Shaking time				
		30 min	60 min	90 min	120 min	180 min
EC (µmhos)	13215	1420	864	712	602	600
TSS (mg/L)	387	220	164	121	111	110
TDS (mg/L)	15556	4750	1250	760	465	460
BOD (mg/L)	8865	1470	910	554	427	420
COD (mg/L)	3125	3040	2110	1750	1450	1400
Chlorides (mg/L)	15111	4900	1681	1110	410	300
Sodium (mg/L)	2440	1140	750	470	425	420
Potassium (mg/L)	1896	1175	750	350	120	100
Magnesium (mg/L)	520	320	214	114	85	75
Calcium (mg/L)	340	220	175	105	75	54
Zinc (mg/L)	35	21	16.5	11.1	8.0	4.5
Chromium (mg/L)	112	58.7	24.2	12.5	5.3	4.1
Nickel (mg/L)	46	21.9	15.5	11.8	10.5	8.5
Copper (mg/L)	55.5	29.5	21.4	17.4	11.5	7.2
Cadmium (mg/L)	7.8	6.1	4.4	2.9	2.5	2.0
Lead (mg/L)	7.5	4.9	4.1	3.1	2.5	2.0

Table 3. Effect of pH on the treatment capacity of CS/PVA/SF (1:1:1) electroplating industry effluent

Parameters	Raw effluent	pH					
		4	5	6	7	8	9
EC (µmhos)	13215	1750	550	602	3110	5112	6120
TSS (mg/L)	387	270	110	111	125	144	176
TDS (mg/L)	15556	5150	440	465	1300	2503	6410
BOD (mg/L)	8865	2570	410	427	1105	2100	2750
COD (mg/L)	3125	2740	1350	1450	1970	2150	2850
Chlorides (mg/L)	15111	4549	310	340	840	4005	5230
Sodium (mg/L)	2440	976	410	471	660	975	1154
Potassium (mg/L)	1896	685	98	122	320	612	753
Magnesium (mg/L)	520	250	75	95	144	215	310
Calcium (mg/L)	340	175	22	35	98	170	225
Zinc (mg/L)	35	16.2	5.0	5.9	10.4	17.5	20
Chromium (mg/L)	112	30.9	4.0	8.1	13.5	46	65
Nickel (mg/L)	46	21.8	5.4	8.9	15.2	24.7	31.5
Copper (mg/L)	55.5	29.8	9.3	15.5	19.1	22.8	37.4
Cadmium (mg/L)	7.8	6.5	2.0	2.9	3.9	5.7	6.9
Lead (mg/L)	7.5	5.7	1.7	2.7	3.6	5.2	6.1