STUDIES ON PHYSIOCHEMICAL, CADMIUM REMOVAL IN DIFFERENT STATIONS OF THAMIRABARANI RIVER BASINS USING GREEN FABRICATION OF SILVER NANOPARTICLES BY CHLOROPHYTA (*ULVA LACTUCA*) AS Cd ADSORBENT

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Abstract

All living things depend on water resources for sustenance, yet as anthropogenic activity has increased, most water bodies have been polluted with chemical and biological contaminants. It incorporates animal waste, pesticides, dyes, hydrocarbons, heavy metals, and more. Such exposures raise the possibility of developing health risks such heavy metal toxicity, diarrhoea, and viral or bacterial infections. The statistical analysis of the physiochemical traits at five different stations revealed that physical circumstances were insignificant ($p< 1.0$) at the 0.05 p level, however chemical components were not significant ($p< 0.9691$) at 0.05 p level. Regarding the concentration of cadmium, virivilai stations exhibited higher amount of 0.171 ± 0.0150 ppm/mL compared with other stations. The concept of adsorption property of metal nanoparticles was brought up by this heavy metal hazards; consequently, NPs has vast surface area to interact with target. In the present research, an ethanolic extract of the chlorophyta (Ulva lactuca) and its silver nanoparticles were used to create a comparative model of Cd adsorption. Ulva AgNPs had more effective adsorption properties than extract alone, according to the results of this comparative Cd adsorption behaviour study. For a better understanding of the Cd adsorption process for heavy metal bioremediation, more standardisation and characterisation has to be performed.

Keywords: Physiochemical parameters, Heavy metal (Cd), Bioremediation, *Ulva lactuca*, *Ulva* AgNPs, Cd adsorption and Atomic absorption spectroscopy (AAS).

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Introduction

Water is frequently seen as an essential requirement for life. Two thirds of the human body is supposedly made up of water. The most significant medium in these ecosystems is water. Effluents, both organic and inorganic, contaminate water. Rapid changes in the population, technology, and industry have led to significant problems and poor environmental conditions. The ability of the industrial sectors to release effluents into bodies of water remains unaffected. There are around 2,000 distinct chemical pollutants, and 750 or so of them are found in drinking water. Minor components include magnesium carbonate and iron oxide. The salt concentration of offshore seawater ranges from 30,000 to 36,000 mg/L of dissolved solids, including 19,000 mg/L of chloride, 10,500 mg/L of sodium, and 1270 mg/L of magnesium.

Fresh groundwater from mountains and brackish sea water has densities that are around 2.5% higher than that of fresh water. After thousands of years of human development, during which water was a bountiful resource that was almost always available for free, the situation is now rapidly changing to the point that water scarcity is emerging, particularly in the driest parts of the planet and becoming the greatest danger to human health, food security, and natural ecosystems. A recent IWMI study (Seckler *et al*., 1998) estimates that 1.4 billion people, or 25 percent of the world's population, or 30 percent of the population in developing countries, reside in regions where there will be severe water shortages in the first half of the century. Contamination of water India's expanding population has led to excessive air and water pollution, as well as deforestation. Water contamination is brought on by point sources, including industrial waste and human and agricultural waste (Amarsinghe & Sharma, 2009).

The water bodies are safe to consume and are not seriously polluted. Few requirements were found in a few of the higher-ranking stations was reported by Sreeja.V. and Ramalingom Pillai (2012) for recommend taking precautionary measures to ensure residential water quality. Heavy metal emissions from human activities, including industrial, mine, and waste disposal, cause soil contamination. These non-degradable metals persist long after release, posing a significant environmental concern (Musilova .J *et al*., 2016). Soils and water bodies are the main environmental compartment used for the disposal of heavy metals caused by anthropogenic activity. As a result of the majority of heavy metals' resistance to microbial or chemical degradation, their levels of toxicity remain for a very long time after they are released into the environment (Lepp N.W., 2012; Athar M., 2001). This article focuses on the physiochemical and heavy metal influences on waters bodies of thamirabarani river at different stations through kanyakumari district.

Materials and Methods

Study Area

The Tamiraparani River Basin, one of 17 river basins, covers the districts of Tirunelveli and Thoothukudi in southern Tamil Nadu. It has 7 sub basins and covers an area of 5717.08 sq. km. The basin is bounded by the Vaippar river basin to the north and Nambiyar and Kodaiyar river basins to the south. It travels through Thickurichy, Pacode, Kuzhithurai, Virivilai, and Mankadu before joining the south-western coast of India, bordering the Arabian Sea to the west.

Collection of Samples

Five separate locations, including Thickurichy (Site-I), Pacode (Site-II), Kuzhithurai (Site-III), Virivilai (Site-IV), and Mankadu (Site-V), were used to collect water samples from the Thamirabarani River for this study project. The water samples were taken by dipping sterile containers into the water and specifying the location for each sampling. The samples were taken, and they were sent right away to the lab for physiochemical and heavy metal analysis.

Analysis of physio-chemical parameter of collected Thamirabarani River Basin

The physical and chemical characteristics of the study samples of Thamirabarani River water from Thickurichy (Site-I), Pacode (Site-II), Kuzhithurai (Site-III), Virivilai (Site-IV), and Mankadu (Site-V) were analysed. These characteristics included appearance, colour, odour, pH, temperature, turbidity, dissolved solids (DS), and electrical conductivity (EC). Using the Standard Protocols of the Tamilnadu Water Supply and Drainage Board, Nagercoil, the following variables were measured: total alkalinity, total hardness, calcium, magnesium, sodium, potassium, iron, manganese, free ammonia, nitrite, nitrate, chloride, fluoride, sulphate, phosphate, and dissolved oxygen.

Analysis of Toxic Heavy metal traces in Thamirabarani River Basin Water Samples

The selected water and soil samples were collected from five different sites (Thickurichy (Station-I), Pacode (Station-II), Kuzhithurai (Station-III), Virivilai (Station-IV) and Mankadu (Station-V) of the Thamirabarani River and processed for further heavy metal analysis. The collected water samples were transferred to the lab and filtered through grade 1 Whatman filter paper. Atomic absorption spectrophotometer (Type Model Name ROM Version S/N) with the parameter of flow rate fuel gas (L/min) was 1.8, support gas flow rate (L/min) was 15.0, Air-C2H2 (Acetylene) flame type and wavelength for cadmium (Cd) is 228.8nm and the concentration was denoted as parts per million (ppm).

Green Synthesis of Ulva Silver Nanoparticles as Cd Adsorbent

Green synthesis of *Ulva* AgNPs was achieved by Garima. S. *et al*., (2011) methodology of taking 0.05 M of aqueous solution of silver nitrate ($AgNO_3$) was prepared and used for the synthesis of silver nanoparticles. 5mL of ethanolic extract of *Ulva sps* was added to 200 mL of 0.05 mM $AgNO_3$ solution for bioreduction process with the agitation using Magnetic stirrer at room temperature. This reduction reaction was enhanced with the dropwise addition of 0.05 M % of Ascobic acid (Vitamin C). The prepared Ag nanoparticles were centrifuged at 3000 rpm for 10 minutes. Wash the pellet with distilled water 2 to 3 times. And the pellet was re-dissolved in sterile distilled water and dried to obtain powered form of Ag Nanoparticles. To study adsorption isotherm, stock solution (100 ppm/mL) were prepared and initial pH of solution was in the range of 2-3 and adjusted to 6-7 using 0.1 N HCl or NaOH solutions. Various concentrations of (20, 40, 60, 80 and 100 µg/mL) of respected samples (Rice husk, Biochar, Ulva extract and Ulva AgNPs) were added to each 15 mL centrifuge tube containing 10 ml cadmium solution. Whole content was agitated on orbital shaker for the time of 15 mins at room temperature, 25°C. After completion of different contact time (0.5, 1, 2, 4 and 8 hours) contents of flask were filtered through whatman filter paper No. 41 and filtrate was analyzed for the residual cadmium concentration in solution by using AAS and represented as ppm/10mL concentration of Cadmium.

Characterization of *Ulva* AgNPs as Cd Adsorbent

The synthesized *Ulva* AgNps additionally characterized for the crystalline property through XRD the range of 20-80 θ (couple two theta/theta) in single crystal x-ray diffractometer – BRUKER Q8 QUEST. The peak data was interpreted and assessed the characteristic features of AgNps and Raman spectroscopy was analyzed at 400 to 1800 $^{cm-1}$ in Confocal Raman Spectroscopy- WITEC ALPHA 300-R under a 785 nm diode laser excitation. The laser power is 5 mW. A microscope with a Leica 20 objective (NA=0.4) and a spectral resolution of 2 cm1 was used to gather spectra in backscattering geometry, and a Peltier-cooled charge-coupled device (CCD) camera was used to detect the Raman signal. For spectrum capture and analysis, the software programme WIRE 2.0 (Renishaw) was used. The integration period for the acquisition of SERS spectra was 10 s. TG/DTA (DTG-60, Shimadzu, Kyoto, Japan) was evaluated for thermal stability and weight loss of the prepared AgNPs at the range of 25 to 800 °C at atmospheric condition.

Statistical Evaluation

The data were obtained from the above experiments of physiochemical and heavy metal corruption of study area was statistically evaluated through Two-way ANOVE, Standard deviation, and Standard errors and plotted respective graphical representations.

Results

Analysis of physiochemical parameters of collected Water samples

After being visually examined for any debris and organic wastes, the obtained water samples appeared to be clear in terms of colour, appear, and odour. **Table: 1.** Higher turbidity was estimated at stations I and V (3 NT units), and as a result, higher TDS levels were measured at stations IV (211 mg/L), I (196 mg/L), and V (193 mg/L). EC was found to be considerable at many Thamirabarani River stations. One-Way ANOVA was employed to assess the overall differences in the physical data at the 0.05 level, and the computed results revealed no statistically significant differences (P value = 1.0) at the 0.05 level. In the Thamirabarani River water samples that were taken, macronutrients like nitrogen, phosphorous, and potassium were discovered to be common, as well as micronutrients like manganese, iron, zinc, and copper, which were calculated in all five stations given in **Table: 2**. Even though water bodies are often used for bathing and watering, there were slight variations in the concentrations of macro and micronutrients that were observed; nonetheless, these variations were not statistically insignificant. The calculated p-value was 0.9691 at p 0.05, indicating that there was not much variation in the data from the five separate sample locations **(Figure: 1)**.

Table: 1. Physical Parameters of Thamirabarani River water in Different Stations

Physical Parameters	Station-I (Thickurichy)	Station-II (Pacode)	Station-III (Kuzhithurai)	Station-IV (Virivilai)	Station-V (Mankadu)
Appearance	Clear	Clear	Clear	Clear	Clear
Colourless	Colourless	Colourless	Colourless	Colourless	Colourless
Odour	-	-	-	-	-
Turbidity (NT unit)	3	2	0	1	3
Total dissolved solid (mg/L)	196	174	163	211	193
Electrical conductivity (Micro mho/cm)	268	285	298	282	279
Temperature (°C)	26.4	27.1	27.8	27.8	28.1
One Way ANOVA	The Physical Parameters of different stations were no significant ($p<1.0$) at 0.05 p level				

Table: 2. Chemical Parameters of Thamirabarani River water in Different Stations

Chemical Paramers	Station-I (Thickurichy)	Station-II (Pacode)	Station-III (Kuzhithurai)	Station-IV (Virivilai)	Station-V (Mankadu)
pH	7.4	7.1	7	7.3	7.5
Total Alkalinity as $CaCO_3$ (mg/L)	38	34	31	46	62
Total Hardness as $CaCO_3$ (mg/L)	89	84	76	74	91
Calcium as Ca (mg/L)	27	22	19	23	29
Magnesium as Mg (mg/L)	9	8	8	11	9
Sodium as Na (mg/L)	27	25	18	19	21
Potassium as K (mg/L)	8	7	3	5	7
Iron as Fe (mg/L)	0.23	0.19	0.17	0.5	0.4
Free Ammonia as NH_3 (mg/L)	0.37	0.31	0.32	0.39	0.41
Nitrite as NO_2 (mg/L)	0.04	0.04	0.02	0.03	0.03
Nitrate as NO_3 (mg/L)	1	1	1	3	5
Chloride as Cl (mg/L)	69	61	47	59	62
Fluoride as F (mg/L)	0.2	0.2	0.2	0.8	0.6
Sulphate as SO_4 (mg/L)	18	17	9	20	19
Phosphate as PO_4 (mg/L)	3.7	3.2	1.2	3.1	3.5
Dissolved Oxygen 4 hrs. as O_2 (mg/L)	0.2	0.2	0.4	0.3	0.2
pH Alkalinity as $CaCO_3$ (mg/L)	-	-	-	-	-
Manganese as Mn (mg/L)	-	-	-	-	-
One Way ANOVA	The Chemical Parameters of different stations were no significant ($p< 0.9691$) at 0.05 p level				

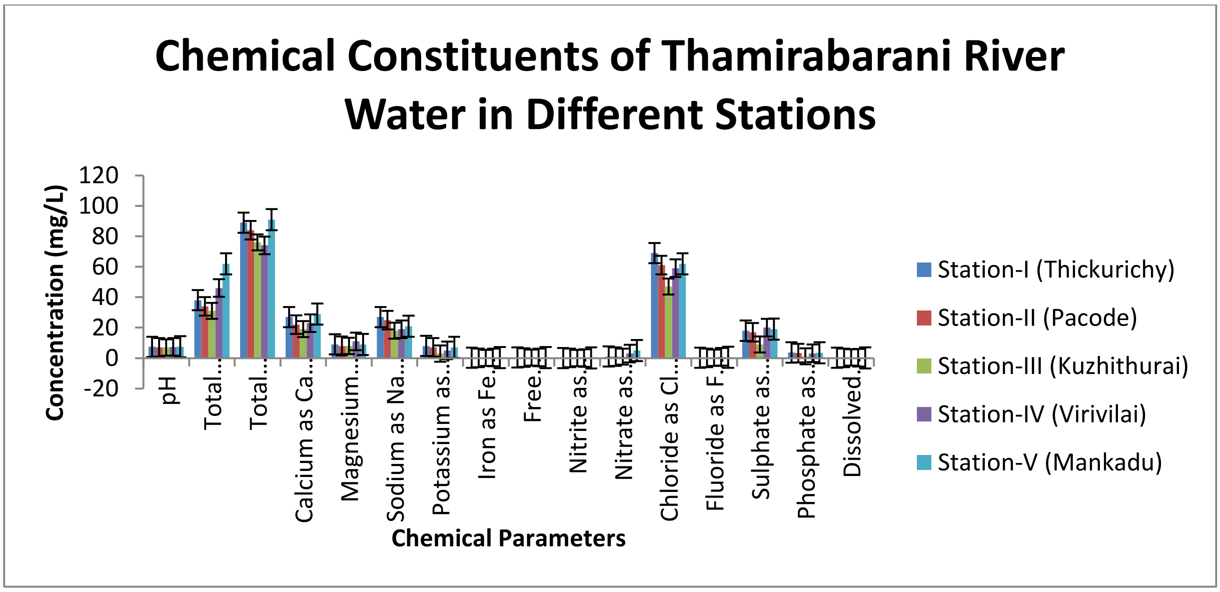

Figure: 1. Chemical Parameters of Thamirabarani River water in Different Stations

Analysis of Toxic Heavy metal traces in Thamirabarani River Basin Water Samples

Atomic adsorption spectrophotometry was applied to assess the concentration of the heavy metal (cadmium) in the water samples from the five distinct stations of the Thamirabarani river basins. showed that the highest amount of cadmium was found in station-IV (Virivilai), which measured around 0.171 ± 0.0150 pmm/ mL, followed by 0.163 ± 0.0142 ppm in Kuzhithurai (station-III), 0.16 ± 0.0032 ppm in Pacode (station-II), 0.159 ± 0.0028 ppm in Thickurichi (station-I), and 0.149 ± 0.0032 in Mankadu respectively showed in **(Figure: 2)**.

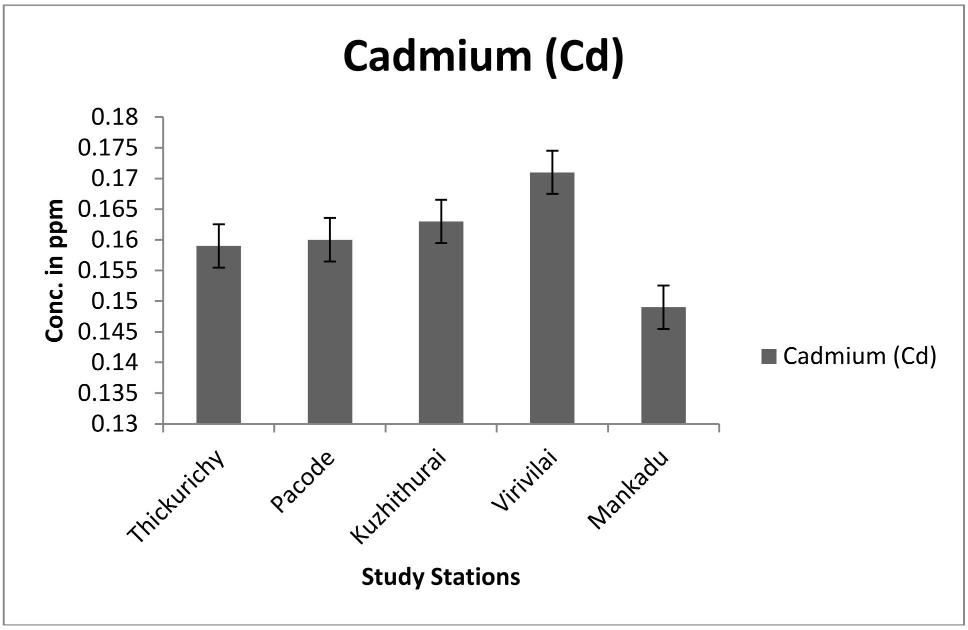

Figure: 2. Estimation of Cadmium (Cd) of Thamirabarani River water in Different Stations

Synthesis of AgNPs as Cd Adsorbent

Silver nanoparticles was prepared using chlorophyta (*Ulva luctus*) ethanolic extract by soxhlet method and the crude ethanolic has later concentrated using rotary evaporator dissolved with distilled water. The yield of *Ulva* AgNps was quantified as 3.38 g in 200 mL along with 5 mL of Extract and employed for Cd adsorbent using AAS. After synthesis, the *Ulva* AgNPs was subjected to Cd adsorption potential with different concentration and contact time. According to the Cd estimation by AAS, the maximum cadmium adsorption (41.2 ± 0.10 ppm/mL) was attained at the concentration of 100 (µg/mL) of AgNPs and the dosage of Cd in test solution decreases with increased condition of AgNPs adsorption capacity comparatively. Although, the contact time is another parameter influences the adsorption of Cd in AgNPs suspension. Tabulated data **(Table: 3)** indicated that the exposure time increases, the concentration of Cd decreased in test sample 39.14 ± 0.04 ppm/mL for 8 hrs showed in **Figure: 3a and b**.

Table 3: Concentration of Heavy metals in Prepared Adsorbent Solutions

Cd 100 ppm in 10 mL	**Cd Concentration (ppm/10 mL)**				**Adsorbant Conc. (100 µg/mL)**
Concentration (µg/mL)	**Ulva Extract**	**Ulva AgNPs**	**Ulva Extract**	**Ulva AgNPs**	**Time (Hours)**
20	97.51 ± 0.10	89.08 ± 0.07	95.08 ± 0.05	41.09 ± 0.05	**0.5**
40	97.21 ± 0.10	75.92 ± 0.04	94.91 ± 0.02	40.72 ± 0.05	**1**
60	96.85 ± 0.05	61.72 ± 0.04	94.51 ± 0.05	40.33 ± 0.05	**2**
80	96.4 ± 0.03	54.82 0.04	94.14 ± 0.05	39.88 ± 0.07	**4**
100	95.87 ± 0.05	41.2 0.10	93.93 ± 0.05	39.14 ± 0.04	**8**
One Way ANOVA	(p> 0.0047) at 0.05 p level		(p< 1.05) at 0.05 p level		**One Way ANOVA**

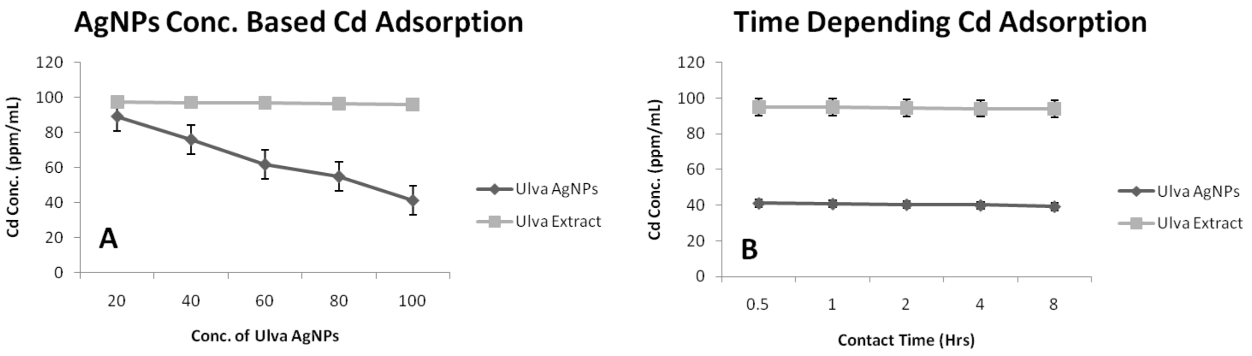

Figure: 3.a) Cd adsorption with Ulva AgNPs Concentration; b) Contact time depending Cd Adsorption

Characterization of *Ulva* AgNPs

The XRD pattern of synthesized *Ulva* AgNPs was evaluated the plane of the silver crystals was ascribed to the XRD peaks 32.164°, 38.030°, 44.203°, 64.394°, and 77.297°, respectively, indicating that the silver nanoparticles are face-centered, cubic, and crystalline in nature **(Figure: 4.a)**. Silver nanoparticles' "rough surface" might act as "hot sites" for surface plasma. With a rougher surface, the silver nanoparticles exhibit higher Raman enhancement. It is significantly influenced by the density of silver nanoparticles, according to the results **(Figure: 4.b)**. **Figure 4.c** shows the TGA and DTA curves of powder Ulva silver nanoparticles. The TGA curve shows that the AgNPs lost the majority of its weight in the temperature range between 100 and 200°C. Nearly little weight loss occurs between 100 and 300 degrees Celsius. It is commonly believed to be caused by the evaporation of water and organic substances. Overall, the TGA data indicate an 84% loss over 300°C. The major cause of the DTA plot's high exothermic peak between 200°C and 300°C is the crystallisation of silver nanoparticles. DTA profiles demonstrate synchronous full thermal breakdown and crystallisation of the sample.

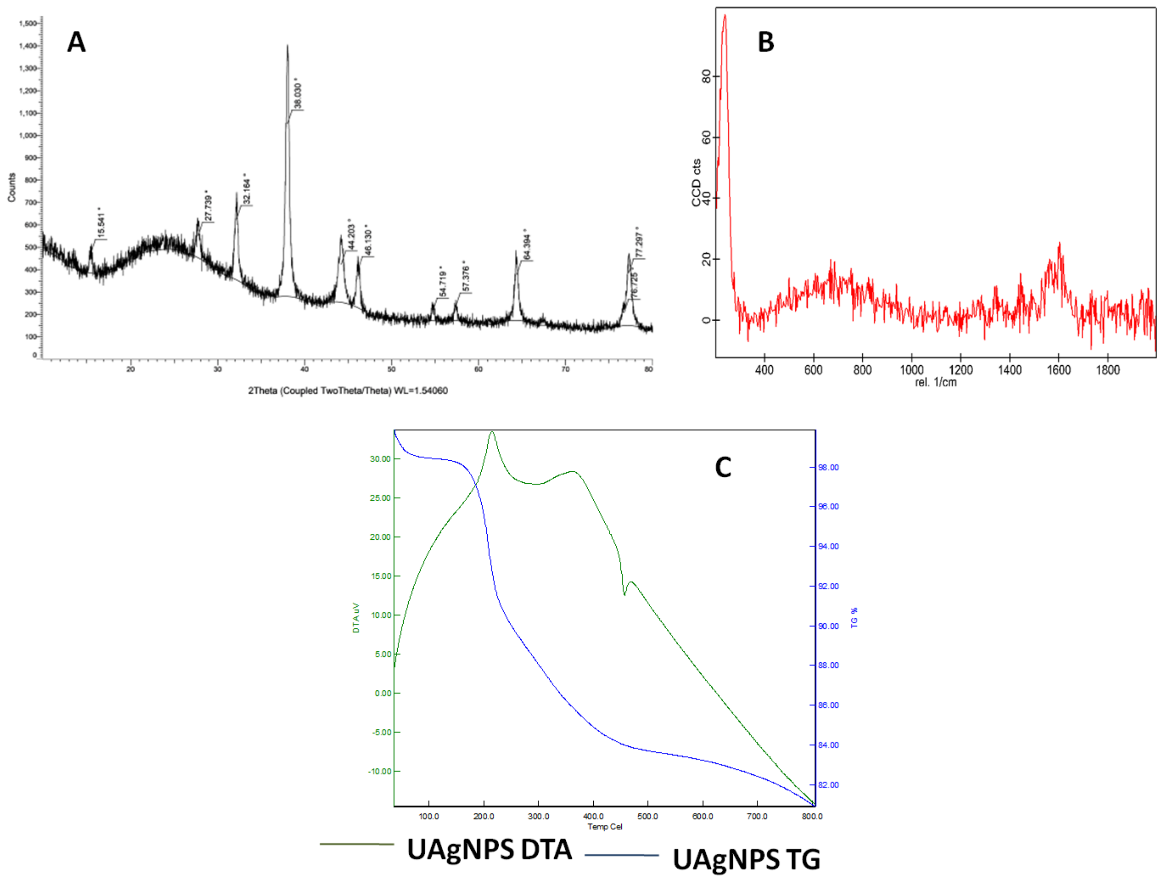

Figure: 4. a) XRD Pattern of Ulva AgNPs; b) Raman Spectra of Ulva AgNPs; c) TG/DTA property of Ulva AgNPs

Discussion

Water pollution is primarily measured by assessing the physico-chemical parameters of water, which has now become a crucial step in the conservation of aquatic resources and ecosystems (Latha and Mohan, 2010). High chloride concentrations are thought to be a sign of contamination from organic animal wastes, which are bad for aquatic life and problematic in irrigation water (Rajkumar *et al*., 2004). Total hardness is a measure of the water's quality that quantifies the impact of dissolved minerals (Ca2+ and Mg2+), which determines the water's solubility for residential, industrial, and drinking purposes due to the existence of bicarbonates, sulphates, chlorides, calcium, magnesium and nitrate (Arya and Gupta, 2013). A higher Mg content makes the water tasteless and acts as a laxative on humans (Preeti *et al*., 2009). According to the physiochemical outcomes of the five different stations of thamirabarani river water samples were indicated the agreeability of water quality and usage for farming purposes compared with the standards of BIS and TWAD board.

An element having an exceptionally high atomic weight and a density five times greater than that of water is referred to as a heavy metal (Banfalvi G., 2011). Heavy metals are among the pollutants that are most harmful to the environment, and environmental chemists have concentrated their research on them because of their high level of toxicity. Even while heavy metals are frequently present in trace amounts in natural streams, many of them can still be harmful in large quantities (Herawati N., 2000). The most common heavy metals (Cu) include copper, lead (Pb), nickel (Ni), chromium (Cr), cadmium (Cd), arsenic (As), mercury (Hg), zinc (Zn), and nickel (Ni). Despite the fact that it may only be present in trace levels in the environment, it may seriously harm both human and other animals' health (Herawati N., 2000). Because most heavy metals cannot be wiped out by microbial or chemical activity, their toxicity level lasts for a very long time after they are released into the environment. Humans, who are at the top of the food chain, eat the greatest quality of feed. They are more susceptible to serious health problems as a result of the heavy metal pollution of water sources (Lepp N.W., 2012: Athar M., 2001 & Lee G *et al*., 2002). **Kero Jemal** et al., (2017) was reported that XRD pattern shows crystalline, spherical silver nanoparticles in leaf and callus extracts, with peaks (38.2°, 44.1°, 64.1°, and 77.0°) corresponding to silver crystal planes. Metallic nanostructures have significant effect on the SERS enhancement. It is well known that Raman enhancement is associated with plasmonic “hot spots”, which occur near the contact point of metallic nanoparticles or nanogaps between two or more particles (J. Zhang *et al*., 2005). Particles are mostly spherical and rod in shape, which easily appear as “rough surface,” such as edges, corners, and protuberances (M. Potara *et al*., 2009). Resulted by Majeed Khan *et al*., (2011) from TGA/DTA supported the exothermic process brought on by the desorption of chemisorbed water as well as the weight loss. When the resistivity of a thin layer of silver is measured throughout a temperature range of 100 to 300 K, the sample exhibits semiconducting behaviour.

Conclusion

According to the findings of ohysiochemical and heavy metal screening of collected water samples in five different stations of thamirabarani river basins exhibited significant to the standards followed by BIS and TWAD board. This was further directed to manage the use of this water bodies in house hold purposes by treating with different adsorbents for the removal of heavy metal (cadmium) modified with classical filteration system.

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