# A COMPARATIVE STUDY OF HEAVY METALS (Cd & Hg) IN GROUND FRESH WATER, SEWAGE WATER AND BAEN NALA WATER (SHAKARGARH) AND THEIR ACCUMULATION IN VEGETABLES IRRIGATED BY THESE WATER SOURCES (A CASE STUDY)

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*ABSTRACT: The detection and determination of heavy metals (Cd & Hg) was made between different irrigation sources like fresh ground water, sewage water and Baen Nala water in the rural areas of district Narowal and finally the comparison of the vegetables grown by these different irrigation sources was made. Five vegetables (i.e. Turnip, radish, coriander, spinach and brassica) were selected. The Atomic Absorption Analysis was made that showed the concentration of Cadmium and Mercury was comparatively high in leafy vegetables such as spinach and brassica. The Cadmium concentration was higher than the permissible limits of WHO i.e. 0.1 mg/kg in leafy vegetables like spinach and brassica leaves. However, the Mercury concentration in both vegetables and soils were found within the permissible limits given by WHO/ FAO (1999) and safe in consumption point of view.*

*Key Words: Cd & Hg accumulation, irrigation sources & vegetables, Areas of District Narowal, WHO/FDA limits.*

## INTRODUCTION:

There is a very vital role of vegetables because they balance the human food in the same way as Allah almighty has produced vegetables and all important ingredients are included in them such as carbohydrates, proteins, vitamins, minerals and trace elements. Vegetables play an important role in human diet as it contains necessary ingredients required for human health [1]. The increased usage of vegetables in human diet may cause the accumulation of toxic metals in human body as the vegetables may contain both essential and toxic elements. Many heavy metals accumulate in vegetables over a wide range of concentrations from water, soil, manure and the chemicals which are used to increase their production and to kill harmful insects [2].

The use of sewage water to irrigate the soils has been widely practiced for many years. The sewage water abundantly consists of both organic and inorganic compounds that play an important role in plants growth [3,4]. The sewage water is a cheaper source of irrigation and is easily available to the farmers near discharged areas outside the cities because many cities discharge their wastes without proper treatment and so this water may contain many toxic elements and when this sewage water is used to irrigate the fields, these toxic metals accumulate in vegetables especially in their edible parts. Sewage Water has been applied to agricultural soils, forest soils and land disposal sites of many countries around the world [5, 6] and also same practice is performed in District Narowal.

According to the report published in daily newspaper The Dawn in 2006 approximately more than hundred thousand million gallons of sewage is being produced per day from main cities like Lahore, Kasur, Faisalabad, Karachi, Multan, Rawalpindi, Peshawar, Hyderabad, Sukkhar, Quetta and is used to irrigate approximately 30000 hectares of agriculture land [7].

Sewage water contains heavy amount of metals like Cd, Hg, K, Pb, Na, Cr, Cu, Ni, Zn, Co & Mg, K. Toxicity of heavy metals has attracted global attention due to their mutagenicity, teratogenicity, carcinogenicity and related impacts on living systems [8, 9]. The use of heavy metals for a long time through diet may affect the human kidney and liver and may lead to heart diseases and nervous problems [10, 11].

### Permissible limits

The limiting concentration of Cadmium by NEQS (National Environmental Quality Standards) for irrigation water is 0.10 µgm/L. United Nations standard for nutritional and agricultural materials has set the maximum permissible level of 0.02 ppm for Cd content in irrigational waters [12]. According to WHO maximum permissible limit for Cadmium content in food products is 0.1 µg/g [13]. A permissible limit for Hg in irrigation water as mentioned by NEQS (National Environmental Quality Standards) is 0.01mg/L [14, 15]. The maximum permissible limit of 0.03µg/g has been specified by WHO/ FDA [16] and ATSDR in foods and food stuff. The critical level of mercury in solid and liquid food which is permissible is 0.5 ppm.

## MATERIAL AND METHODS:

### Instruments

### Atomic Absorption Spectrometer

### MHS-15 Mercury Hydride Generator

Mercury was analyzed by Perkin Elmer MHS-15 as Mercury Hydride Generator coupled with AAnalyst 700 atomic absorption spectrometer. It gives a detection limits up to very low ranges.

### Graphite Tubes

Pyrolytic graphite were used that provided STPF (Stabilized Temperature Platform Furnace) conditions.

### Hollow Cathode lamp for Cadmium

As line source Hollow cathode lamp having wavelength 228.8 nm was used. The setting was automatically set by the instrument software.

**Electrodeless discharge lamp for Mercury**

For Mercury Perkin Elmer Coded EDL with built in power supply having wavelength 253.7nm was used.

**Analytical Balance**

Shimadzu AUX 200 micro analytical balance capable of weighing 10 mg to 220 mg.

**Electric oven**

Carbolite Electric Oven used for drying soil and vegetable samples.

**Hot Plates**

STEDEC Rectangular Hot Plates (Temperature range 300 - 350℃ ).

**Materials & Apparatus**

**Auger sampler**

A standard stainless steel auger hand sampler for sampling at the depth of 10 cm of soil.

**Polythene bags**

Polythene bags were used for the preservation of soil and vegetables samples.

**PTFE Bottles (1.5 L)**

PTFE bottles were used for the sampling of water samples.

**Grinding and crushing Apparatus for Vegetable samples**

Laboratory disc mill of Tema Corporation UK and Fritsch sieve shaker was used for grinding sieving of soil and oven dried vegetables samples.

**Polythene Gloves**

Polythene gloves were used for the handling samples in order to avoid any contamination of from perspiration from hands samples.

**Glass ware**

Standard Pyrex glassware like beakers, volumetric flasks, funnels etc. was used for preparation and digestion of samples. All laboratory glass wares kept overnight in 10%v/v $HNO_3$ acid solution and rinsed with deionized water and dried in a dust free environment prior to use.

**Chemicals and reagents**

Deionized water

Potassium Permanganate Merck Extra pure

Nitric acid 67% BDH Spectroscopy Grade

Hydrofloric acid 40% BDH Analar Grade

Perchloric acid 70% BDH Analar Grade

Sulphuric acid 98% panreac Spectroscopy Grade

BDH Stock Standard Solutions of 1000mg/L of Cd and Hg for atomic absorption spectrometry

Hydrated Tin Chloride Merck Extra pure

Hydroxylamine sulphate Merck Extra pure

Sodium chloride fluka puriss

Potassium persulphate fluka puriss

**Sampling**

The samples of fresh water, sewage water and Baen Nala water were taken from different areas of district Narowal. Similarly soil and vegetable samples are also collected from different forms irrigated by these three sources. Sampling was done in the month of Jan & Feb 2015.

**Collection of Water Samples**

Water samples were collected in accordance with methods described by EPA in triplicate [17]. The 1.5 L of ground water samples were collected in cleaned and disinfected PTFE bottles from the bores constructed in the vegetable farms for supplying the ground water. The baeen water samples were collected at the points of baeen where waste water is being mixed with it. The sewage water samples were collected directly from outlets of the sewage drains. The collected water samples were acidified with 0.5N Nitric acid to maintain pH<2.

**Collection of Soil Samples**

Soil collection techniques were carried out as described by Radojevic and Bashkin [18]. Soil samples were taken from different corners of each field in triplicate with the help of auger sampler at the depth of 10 cm. 100g of soil was collected from each sampling site. The pebbles & lint in soil was removed with hands covered with polythene gloves. The samples were transferred into properly labeled polythene air tight zipper bags and transferred to laboratory. The soil samples were dried in electric oven, then grinded so that they can pass a 200 mesh sieve. For the analysis of Mercury, soil was not grinded, it only homogenized by using conning and quarting technique.

**Collection of Vegetable Samples**

Five vegetables were selected in which two vegetables (Turnip and Radish) are those whose roots are used as vegetable and three vegetables (Coriander, Spinach and brassica) are those whose leafs are used as vegetable. The vegetables were chosen crosswise from different corners of each field from where soil samples were selected. The vegetable samples are washed with water to remove rotten leaves and deteriorated parts and then transferred to laboratory by packing the samples in sealed polythene zipper bags. In laboratory each type sample was divided into two equal parts. One sample of each type was washed with Type-1 water and the other sample was washed with tap water and considered as unwashed sample and treated as unwashed sample to study that heavy metals are removed on washing or not. Edible parts of vegetables were cut in to small pieces then the samples are dried in air for 3 days. After drying the vegetable, it is grinded then dried in an oven at 110℃ for 20 hours [19]. For the analysis of Mercury the samples were processed as such without grinding.

**Determination of Cadmium**

The determination of Cd was done by using Atomic Absorption Spectrometer. Water samples were analyzed by adopting EPA 3005a [20] method while soil samples were analyzed by using Graphite furnace AAS method [21]. Ganje & Page [22] method was used in present study. Wet ashing acid digestion procedure using mixture of Conc. $HNO_3$, Conc. $H_2SO_4$ and Conc. $HClO_4$ was used.

**Calibration Standards**

1ml of 1000 ppm cadmium chloride solution was taken and total volume was made 100ml by adding Type-1 water.

100ul of stock solution was diluted upto 100ml with Type-1 water for a calibration standard of 10ppb of Cd (Fig. 1).

**Calibration Curve for Cadmium & Mercury**

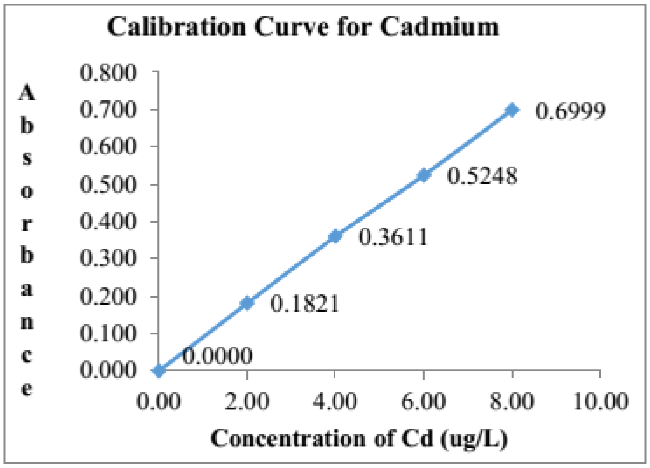


**Figure 1: Calibration Curve of Cd by Graphite Furnace AAS**

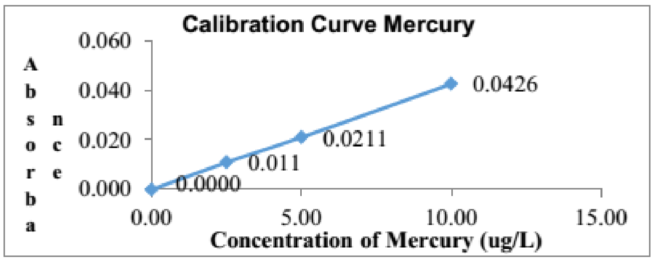


**Figure 2: Calibration Curve of Hg using Perkin Elmer MHS-15.**

**Table 1: Instrumental settings for Cd Analysis.**

| Instrumental Parameter | Setting |
|---|---|
| Wavelength(nm) | 228.8 |
| Slit width (nm) | 0.7L |
| Lamp current (mA) | 4 mA |
| Sample volume (µL) | 20 |
| Signal type: | AA-BG |
| Measuring Signal: | Peak Area |
| Detection Limit(mg/Kg) | 0.001 |
| Injection Temperature | 20°C |

**Table 2: Instrumental Parameters for MHS-15 and AAnalyst700**

| | |
|---|---|
| **Lamp Current** | 185 mA |
| **Analytical wavelength** | 253.6 nm |
| **Slit width and height** | 0.7 nm |
| **Radiation source** | EDL for Hg. |
| **QTA heating** | No flame required. |
| **Prepared measurement volume** | 10 mL |
| **Pre-reaction purge time** | 5 sec |
| **Reaction Time** | 30 sec |
| **Post-reaction purge time** | 50 sec |
| **Carrier Gas Used** | Argon |

## RESULTS AND DISCUSSION

### Cadmium in the water samples

The cadmium content in Sewage water irrigating the vegetable fields in Narowal area was found to be 2.65±0.15 µg/L (Table 3). These values are within the safe limits of NEQS of irrigation water [23]. The cadmium content in Nala baeen water irrigating the vegetable fields near the Nala baeen bridge Shakargarh was found to be 1.15 ± 0.02 µg/L (Table 3). These values are also within the safe limits of United Nation standard for nutritional and agricultural materials [24]. The ground water samples from the tube wells irrigated field showed the lowest Cadmium concentration 1.41±0.01µg/L (Table 3), these values are also within the permissible levels.

**Table 3: Concentration of Cadmium in Water Samples**

| Sample I.D | Cadmium g/L | Mercury g/L |
|---|---|---|
| Sewage water | 2.65 | ND |
| Sewage water | 2.63 | ND |
| Sewage water | 2.62 | ND |
| Ground water | 1.41 | ND |
| Ground water | 1.43 | ND |
| Ground water | 1.39 | ND |
| Baeen water | 1.15 | ND |
| Baeen water | 1.12 | ND |
| Baeen water | 1.11 | ND |

### Cadmium in Soil Samples

Tables show the concentration of Cadmium in soil samples irrigated by different sources of water like ground fresh water (Table 4), sewage water (Table 5) and Nala baeen water (Table 6). The concentration of Cadmium was found to be 0.01 to 0.05 mg/kg, 0.01-0.07 mg/kg and ≤ 0.01 mg/kg respectively. The comparison of vegetable samples indicated in figure-3 clearly shows that the sewage water irrigated soil vegetable of Narowal are more concentrated with Cadmium than the soils of Nala baeen irrigated water and fresh ground water.

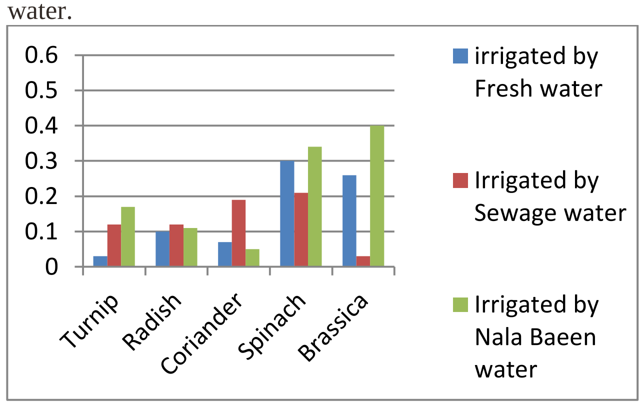


**Figure 3. Comparison of mean Cadmium concentration in vegetables irrigated by Fresh water, Sewage water and nala Baeen water.**

**Table 4: Concentration of Cadmium in soil samples irrigated by fresh water.**

| Sample Identity | Irrigation Water Used | Vegetable Name | Sampling Site | Cadmium *mg/kg | Mercury *mg/kg |
|---|---|---|---|---|---|
| S1 | Fresh Water | Turnip | Kot Nainan | 0.04 | 0.029 |
| S2 | Fresh Water | Turnip | Kot Nainan | 0.05 | 0.025 |
| S3 | Fresh Water | Radish | Kot Nainan | 0.01 | 0.014 |
| S4 | Fresh Water | Radish | Kot Nainan | 0.01 | 0.033 |
| S5 | Fresh Water | Coriander | Kot Nainan | 0.01 | 0.051 |
| S6 | Fresh Water | Coriander | Kot Nainan | 0.01 | 0.028 |
| S7 | Fresh Water | Spinach | Kot Nainan | 0.03 | 0.033 |
| S8 | Fresh Water | Spinach | Kot Nainan | 0.03 | 0.037 |
| S9 | Fresh Water | Brassica | Kot Nainan | 0.01 | ND |
| S10 | Fresh Water | Brassica | Kot Nainan | 0.01 | ND |

*Mean average of three readings · ND =Less than 0.01mg/kg

**Table 5: Concentration of Cadmium in soil samples irrigated by sewage water.**

| Sample Identity | Irrigation Water | Vegetable Name | Sampling Site | Cadmium | Mercury |
|---|---|---|---|---|---|
| S1 | Sewage Water | Turnip | Narowal city | 0.05 | 0.10 |
| S2 | Sewage Water | Turnip | Narowal city | 0.06 | 0.085 |
| S3 | Sewage Water | Radish | Narowal city | 0.01 | 0.043 |
| S4 | Sewage Water | Radish | Narowal city | 0.01 | 0.091 |
| S5 | Sewage Water | Coriander | Narowal city | 0.01 | 0.094 |
| S6 | Sewage Water | Coriander | Narowal city | 0.01 | 0.045 |
| S7 | Sewage Water | Spinach | Narowal city | 0.03 | 0.024 |
| S8 | Sewage Water | Spinach | Narowal city | 0.03 | ND |
| S9 | Sewage Water | Brassica | Narowal city | 0.01 | ND |
| S10 | Sewage Water | Brassica | Narowal city | 0.01 | 0.023 |

*****Mean average of three readings ND =Less than 0.01mg/kg

**Table 6: Concentration of Cadmium in soil samples irrigated by Nala Baeen water.**

| Sample Identity | Irrigation Water | Sampling Site | Vegetable Name | Cadmium | Mercury |
|---|---|---|---|---|---|
| BS-l | Baeen Water | Shakargarh bridge | Turnip | ND | 0.045 |
| BS-2 | Baeen Water | Shakargarh bridge | Turnip | ND | 0.054 |
| BS-3 | Baeen Water | Shakargarh bridge | Radish | ND | 0.01 |
| BS-4 | Baeen Water | Shakargarh bridge | Radish | ND | 0.112 |
| BS-5 | Baeen Water | Shakargarh bridge | Coriander | ND | 0.01 |
| BS-6 | Baeen Water | Shakargarh bridge | Coriander | ND | 0.307 |
| BS-7 | Baeen Water | Shakargarh bridge | Spinach | ND | 0.305 |
| BS-8 | Baeen Water | Shakargarh bridge | Spinach | ND | 0.276 |
| BS-9 | Baeen Water | Shakargarh bridge. | Brassica | 0.01 | 0.215 |
| BS-10 | Baeen Water | Shakargarh bridge | Brassica | 0.01 | 0.284 |

*****Mean average of three readings ND =Less than 0.01mg/kg

## Cadmium in Vegetable Samples

Tables below show results for concentration of cadmium in five vegetable samples i.e. Turnip, Radish, coriander, spinach and brassica leaves irrigated by fresh (Table 7), Nala Baeen (Table 8) and sewage water (Table 9) respectively. The vegetables grown on soils irrigated by fresh water contained cadmium from 0.03-0.33 mg $kg^{-1}$. The leafy vegetables such as spinach showed the highest concentration up to 0.33 mg $kg^{-1}$ which are well above the limits specified by WHO/FDA that is 0.l mg $kg^{-1}$. The vegetables grown on soils irrigated by Nala baeen water contained cadmium from 0.03-0.22 mg $kg^{-1}$. The spinach showed the highest concentration up to 0.22 mg $kg^{-1}$ while turnip contained the concentration as low as 0.03 mg $kg^{-1}$. The vegetables grown on soils irrigated by sewage water contained cadmium from 0.03-0.41 mg $kg^{-1}$. The brassica leaves showed the highest concentration up to 0.41 mg $kg^{-1}$ while turnip contained the concentration as low as 0.13 mg $kg^{-1}$. It seems that cadmium uptake is more in the vegetables irrigated by sewage water and nala baeen water respectively as compared to those irrigated by fresh ground water.

**Table 7. Concentration of Cadmium in vegetable samples irrigated by fresh water.**

| Sample Identity | Name of Vegetable | Irrigation Water | Sampling Site | Cadmium *mg/Kg | Mercury *mg/Kg |
|---|---|---|---|---|---|
| V-1 | Turnip | Fresh Water | Kot Nainan | 0.03 | ND |
| V-2 | Turnip | Fresh Water | Kot Nainan | 0.03 | ND |
| V-3 | Radish | Fresh Water | Kot Nainan | 0.10 | ND |
| V-4 | Radish | Fresh Water | Kot Nainan | 0.10 | ND |
| V-5 | Coriander | Fresh Water | Kot Nainan | 0.08 | ND |
| V-6 | Coriander | Fresh Water | Kot Nainan | 0.06 | ND |
| V-7 | Spinach | Fresh Water | Kot Nainan | 0.27 | 0.02 |
| V-8 | Spinach | Fresh Water | Kot Nainan | 0.33 | 0.02 |
| V-9 | Brassica | Fresh Water | Kot Nainan | 0.25 | ND |
| V-l0 | Brassica | Fresh Water | Kot Nainan | 0.30 | ND |

*****Mean average of three readings ND =Less than 0.01mg/kg

**Table 8. Concentration of Cadmium in vegetable samples irrigated by Nala Baeen water.**

| Sample Identity | Name of Vegetable | Irrigation Water | Sampling Site | Cadmium *mg/Kg | Mercury *mg/Kg |
|---|---|---|---|---|---|
| V-1 | Turnip | Baeen Water | Shakargarh bridge | 0.06 | ND |
| V-2 | Turnip | Baeen Water | Shakargarh bridge | 0.12 | ND |
| V-3 | Radish | Baeen Water | Shakargarh bridge | 0.13 | ND |
| V-4 | Radish | Baeen Water | Shakargarh bridge | 0.12 | ND |
| V-5 | Coriander | Baeen Water | Shakargarh bridge | 0.16 | ND |
| V-6 | Coriander | Baeen Water | Shakargarh bridge | 0.22 | ND |
| V-7 | Spinach | Baeen Water | Shakargarh bridge | 0.17 | ND |
| V-8 | Spinach | Baeen Water | Shakargarh bridge | 0.22 | ND |
| V-9 | Brassica | Baeen Water | Shakargarh bridge | 0.03 | ND |
| VR- 10 | Brassica | Baeen Water | Shakargarh bridge | 0.03 | ND |

*****Mean average of three readings ND =Less than 0.01mg/kg

**Table 9. Concentration of Cadmium in vegetable samples irrigated by sewage water.**

| Sample Identity | Name of Vegetable | Irrigation Water | Sampling Site | Cadmium *mg/Kg | Mercury *mg/Kg |
|---|---|---|---|---|---|
| V-1 | Turnip | Sewage Water | Narowal city | 0.03 | ND |
| V-2 | Turnip | Sewage Water | Narowal city | 0.17 | ND |
| V-3 | Radish | Sewage Water | Narowal city | 0.08 | ND |
| V-4 | Radish | Sewage Water | Narowal city | 0.14 | ND |
| V-5 | Coriander | Sewage Water | Narowal city | 0.02 | ND |
| V-6 | Coriander | Sewage Water | Narowal city | 0.08 | ND |
| V-7 | Spinach | Sewage Water | Narowal city | 0.25 | 0.02 |
| V-8 | Spinach | Sewage Water | Narowal city | 0.39 | 0.02 |
| V-9 | Brassica | Sewage Water | Narowal city | 0.40 | ND |
| V-10 | Brassica | Sewage Water | Narowal city | 0.41 | ND |

*Mean average of three readings ND =Less than 0.01mg/kg

**Mercury**

**Mercury in water samples**

Mercury concentration in fresh water, sewage water and Nala baen water is shown in Table 3. The concentration of mercury in water was below the permissible limit i.e <2.5 ug/L.

**Mercury in Soil samples**

Table 4,5 & 6 shows the concentration of mercury in soil samples irrigated by fresh water, sewage water and Nala Baeen water respectively. The results showed that most of the soil samples irrigated by the nala baeen water, sewage water and fresh water contained mercury concentration 0.045-0.284 mg/kg, 0.023-0.10 mg/kg and 0.014-0.051 mg/kg respectively. The comparison of vegetables samples depicted in figure 4 clearly shows that vegetables irrigated by Nala Baeen water seem to be more concentrated with mercury than the vegetables irrigated by sewage water and ground water despite the fact that the mercury content of sewage water, Nala Baeen water and fresh water were below the permissible safety standards. Thus atmospheric deposition of the mercury seems also to be contributing to the accumulation of mercury in soil in addition to the water of irrigation used.

**Mercury in Vegetable Samples**

Results for concentration of mercury in five vegetables i.e. Turnip, Radish, Spinach, Coriander and Brassica leaves irrigated by fresh water, Nale Baeen water and sewage water respectively are shown in tables 7, 8 and 9.

This study showed that mercury content was below the detection limit i.e. 0.01mg/kg in all the vegetables except the spinach irrigated by fresh water and Nala Baeen water that contained 0.020 mg/kg thus the mercury content in all the vegetable samples collected in triplicate was within permissible safety limits of WHO/ FDA.

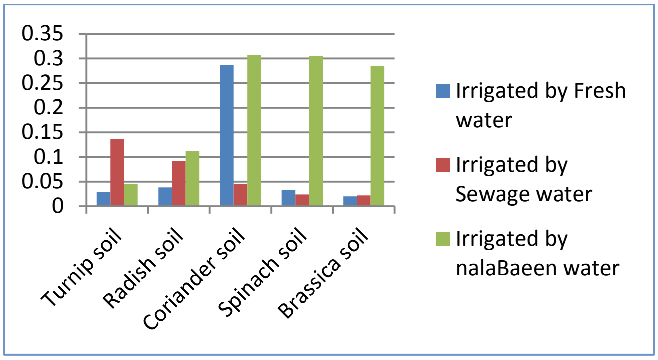


**Figure No 4: Comparison of mean mercury concentration in vegetables irrigated by Fresh water, Sewage water and nala Baeen water.**

## CONCLUSION

Objective of this study was to monitor the level of toxic heavy metals (Cd & Hg) in vegetables with respect to the quality of water used for irrigation Objective of this study was to monitor the level of toxic heavy metals (Cd & Hg) in vegetables with respect to the quality of water used for irrigation, in particular, the sewage water being used for vegetable growth in Different areas like periphery of Narowal, Nala Baeen bridge Shakargarh and Kot Nainan.

Present study shows that the concentration of cadmium, mercury and Arsenic were comparatively high in leafy vegetables such as spinach and brassica. The comparison of cadmium concentration was higher than the permissible limits of WHO i.e. 0.1 mg/kg in leafy vegetables like spinach and brassica leaves. However, the mercury concentration in both vegetables and soils were found to be within the permissible limits given by WHO/ FAO (1999) and safe in consumption point of view. The concentration of Arsenic is also found to be higher than the permissible limits.

The results have revealed that the sewage water causes more cadmium accumulation to the soils and vegetables as compared to the Nala Baeen and fresh water of the tube well bores.

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