TREATMENT OF DEPLETED URANIUM CONTAMINATION IN SOIL BY USING SODIUM BICARBONATE SOLUTION

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ABSTRACT

The Depleted Uranium contamination in soil was treated with chemical leaching method by using sodium bicarbonate with respect to the effect of several variables (Time, Temperature, Bicarbonate Concentration, Carbonate/Bicarbonate Ratio, Oxidative Reagent Effect, pH, Soil/Solution Ratio and Rinsing Effect after treatment) in order to decontaminate or remove Depleted Uranium to acceptable regulatory levels.

The objective is to reach a selectively extracted Depleted Uranium by using a soil washing/extraction without generating a secondary waste which would be difficult to manage and/or dispose off. Results of Depleted Uranium removal efficiency were ranged from (35.4-88.25) %.

الخلاصة

التلوث الناجم عن اليورانيوم المنضب في التربة عولج بإستخدام طريقة الغسل الكيمياوي بواسطة بيكاربونات الصوديوم أخذاً بنظر الإعتبار تأثير متغيرات عديدة (الزمن، درجة الحرارة، تركيز البيكاربونات، نسبة الكاربونات الى البيكاربونات، تأثير العامل المؤكسد، الأس الهيدروجيني(pH)، نسبة التربة الى المحلول، تأثير الشطف بعد المعالجة) لازالة او معالجة التلوث الناجم عن اليورانيوم المنضب وصولا الى نتيجة تكون ضمن الحدود المسموحة.

الهدف كان ايضا انتقاء طرقة معالجة بالغسل الكيمياوي لا تولد تلوثا ثانويا كبيرا يصعب التخلص منه.نتائج كفاءة الأزالة تراوحت ما بين (٣٥،٤ الى ٨٨،٢٥)%. بالإضافة الى دراسة تأثير ثلاثة محاليل(حامض الكبريتيك، حامض الستريك وبيكاربونات يتبعها حامض الستريك) في عملية المعالجة، وكذلك دراسة تاثير المعالجة بالإذابة على(الحديد و الرصاص) في التربة.أخيراً، تم إيجاد علاقات رياضية خاصة بظروف التجارب التي اجريت ما بين تركيز اليورانيوم المنضب والمتغيرات المذكورة أعلاه.

INTRODUCTION

When measuring isotopic ratios in environmental samples it is important to realize that uranium may sometimes become depleted (or enriched) in some of its isotopes due to natural processes such as chemical weathering. Depleted uranium (DU) is a by-product from the process used to enrich natural uranium ore for use as fuel in nuclear reactors and nuclear weapons.

In weapon use ,when penetrator impact on ground surface ,a portion of its DU mass is transformed into aerosols or fine particles and thrown into the surrounding air .These aerosols and fine particles are normally depleted in measurable quantities on the surroundings ground or on other surfaces within about 100m from impact[1].

It is important to solve DU contamination problem in soil. There are several methods of treatment, basically are classified as physical and chemical, soil washing in a conventional sense is based on a physical separation such as screening classification (separation of soil particulate according to their settling velocities). Chemical extraction processes characteristically used to remove uranium from uranium ores are either acid or carbonate based extractions.

For acid extraction, sulfuric acid, which is less expensive than nitric, is the most commonly used acid. Other chemical extractions like carbonate extractions are highly selective for uranium.

The efficiency of the carbonate extractions is based on the formation of sodium or ammonium uranyl tri- and di-carbonate [$UO_2\ (CO_3)_2$ and $UO_2\ (CO_3)_3$], highly stable, water-stable and anionic complexes. Oxidants such as potassium permanganates may be used to increase the extraction efficiency. [2]

In the present work of treatment of DU contamination various variables were examined in leaching experiments such as time, temperature, soil to solution ratio, pH, $NaHCO_3$ concentration, carbonate/bicarbonate ratio, oxidizing reagent and rinsing effect, finally best conditions were obtained to give the best treatment.

- ALKALINE LEACHING OF URANIUM

Alkaline leaching is used only for materials, such as carbonates, which would consume a wasteful amount of acid. Its main advantage is the relatively non-corrosive properties of the solutions employed and the fact that few impurities are dissolved along with the uranium [3].

From these solutions:

1. Carbonate leaching involves the formation of various highly soluble U-carbonate complexes which are not likely to absorb to negatively charged soil constituents [4]. This allows high concentration of uranium in the leachate solution [5].

Some of the key reactions in this process are:

$$CO_3^{-2} + UO_2^{+2} \rightarrow UO_2CO_3 \qquad (1)$$

$$2CO_3^{-2} + UO_2^{+2} \rightarrow UO_2(CO_3)_2^{-2} \qquad (2)$$

$$3CO_3^{-2} + UO_2^{+2} \rightarrow UO_2(CO_3)_3^{-4} \qquad (3)$$

$$6CO_3^{-2} + 3UO_2^{+2} \rightarrow (UO_2)_3(CO_3)_6^{-6} \qquad (4)\ [6]$$

(Mason et al., 1997)[7] Used 0.5M sodium bicarbonate as the dominant reagent, was able to achieve uranium removal of (75-90%).

(Duff. et al., 1997)[8] Found that 0.5M sodium bicarbonate with oxidative compound indicated the overall efficiency of removal (52%) for different soil samples.

(Francis et al., 1994)[9] in their leaching design, used carbonate leaching media showed that >90% of the uranium can be removed from their soil samples.

(Mattus et al., 1993)[10] Found that carbonate extractions generally removed (40-90%) of the uranium from different soil samples.

(Timpson et al., 1994)[11] used the ultrasound treatments combined with carbonate extractions; the result was much as 90% of uranium removed.

(Francis et al., 1997)[2]Utilized carbonate/bicarbonate solution in the treatment, and this solution gave them good results in the removal of uranium.

(Elles et al., 1993)[12] also used the carbonate/bicarbonate solution in the removal of uranium from contaminated soil.

EXPERIMENTAL WORK

- Soil Preparation

The soil samples were dried by exposing to air for four days [13] with respect to good save conditions. After drying soil samples, the impurities (like small metal pieces and plants) were removed and then crushed and sieved (2mm Diameter) [14,15] before the treatment ,the samples were left for (28-30) day to reach the equilibrium state for the radionuclides that exist in soil [13,16].

- Equipment and Materials

1. Beakers & Plastic cups.
2. Filtration Paper. (Whatman 41).
3. Distilled Water.
4. Sensitive Balance (Mettler AC 100).
5. pH Meter (Expandable Ion Analyzer EA B40).
6. Thermometer.
7. Water Bath (Labsco, Germany).
8. Mechanical Compressing Device (Wabash, made in USA)

- Solutions Preparation

Solutions that used for uranium leaching process were prepared as below:

Bicarbonate solution

The bicarbonate solution was prepared with different range of molarities (0.2, 0.3, 0.5, 0.7 and 0.8). The weight of bicarbonate powder was taken by using the sensitive balance, and calculated by using the following equation (for 1liter of distilled water):

M = weight of compound (w)/molecular weight of compound (M.W)

The results were tabulated in **Table (1).**

Table (1):$NaHCO_3$ preparation and pH value.

Weight (gram)	Concentration (molar)	pH
16.8	0.2	8.2
25.2	0.3	8.1
42	0.5	8.06
58.8	0.7	8.02
67.2	0.8	8.03

Each of these weights were diluted with 1 liter of distilled water, then were placed in bottles and labeled with solution name, preparation temperature and date of preparation. The other solutions were prepared in the same manner by using the molarity & normality equations for preparation.(for 1 liter of distilled water)

Normality = Weight of compound (W)/equivalent weight of compound/volume

eq.w= Molecular weight (M.W)/n

Where n = number of protons (in acid-base reaction), or total change in oxidation number of compound (in redox).

Table (2) below shows the solutions, concentrations and pH

Table (2): Solutions preparation

Solutions	Concentrations	pH
Carbonate/Bicarbonate	37 g carbonate+18.5 g bicarbonate/liter	9.03
Carbonate/Bicarbonate	18.5 g carbonate+37g bicarbonate/liter	9.03
Carbonate/Bicarbonate	18.5 g carbonate+18.5 g bicarbonate/liter	8.45

- **Leaching Process**

The three samples were detected and the highest concentration was used in leaching experiments (detection process will be explained later).

All experiments were prepared in the same manner, where 0.8 g of soil were added to batch leaching solution then the solution-soil combination were left for a period. The soil samples solution were filtered by filtration paper, then the samples were dried, prepared to detection procedure and labeled with sample name and other experiment conditions.

A. Time Set

S1 were used in all experiments, because of its high concentration compared with the other samples. This set consisted of five samples, each sample was represented a period of leaching.

The five experiments in this set were prepared as follows:

Soil sample were placed in the plastic cup, then 0.5M of $NaHCO_3$ were added with 1:20 soil to solution weight ratio. This batch solution was left for 2,3,4,5 and 7 days. (at temperature = 15 °C).

B. Temperature Set

Soil sample were placed in plastic cup with 0.5M $NaHCO_3$, soil to solution weight ratio was 1:20, the sample left in flask in water bath for 3 hours and half, this procedure were repeated five times, each one represented a temperature degree (20, 30, 40, 50 and 60 °C).

C. Concentration Set

In this set five molar values have been taken (0.2, 0.3, 0.5, 0.7 and 0.8).Soil samples were used with each of the five solutions for 4 days, at $23C^o$, soil to solution weight ratio 1:20, then the samples were filtered, dried and labeled as the previous sets.

D. Carbonate/Bicarbonate Weight Ratio Set

The experiments are the same, except the experiment conditions were at temperature= 26 °C)., soil to solution ratio = 1:30, the samples were labeled with respect to the solutions' preparation (with Carbonate/Bicarbonate Ratio =1:1, 1:2 and 2:1).

E. pH Set

A 0.5M $NaHCO_3$ was placed in 4 plastic cups, soil samples were added to each one. The pH value were varied by adding NaOH and HCl, pH value were adjusting by using pH meter. **Table (3)** shows the samples' preparation conditions.

Table. (3):pH set solutions' preparation.

Samples	Solutions Composition	pH
S1 pH1	0.5M $NaHCO_3$+3 drops HCl	6.53
S1 pH2	0.5M $NaHCO_3$	8.06
S1 pH3	0.5M $NaHCO_3$+0.25g NaOH	9.01
S1 pH4	0.5M $NaHCO_3$+0.5g NaOH	10.43

Experiments conditions were: temp. =31 °C, soil to solution ratio=1:30, then the samples were left for 2 days.

F. Oxidative Reagent Set

Potassium Permanganate $KMnO_4$ was added to the batch solution (0.5M $NaHCO_3$) of three soil samples (0.8 g) under the conditions temperature=25 °C, soil to solution ratio=1:30) and left for 2 days of leaching, the samples and its solution were prepared as shown in **Table(4)**:

Table (4):Oxidative reagent set solution preparation.

Samples	Batch Solution
S1O1	0.5M $NaHCO_3$+0.016 g $KMnO_4$
S1O2	0.5M $NaHCO_3$+0.024 g $KMnO_4$
S1O3	0.5M $NaHCO_3$+0.032 g $KMnO_4$

It was important to study the oxidation effect on the treatment, so in the same condition but without adding $KMnO_4$, sample S1bo was prepared.

G. Soil to Solution Ratio Set

In this set five different soil to solution ratios were prepared by using (0.5M $NaHCO_3$) at 19 °C (Soil/ Solution Ratio=1:10, 1:20, 1:30, 1:40, 1:50), The samples were left for 2 days in the leaching solution.

H. Rinsing Effect

Distilled water were used to enhance the treatment process, two experiments were made; treatment with 0.5M $NaHCO_3$ followed by one rinsing (2 days left in distilled water) and by two rinsing (4 days left in distilled water), the sample before rinsing were prepared at 26 °C, with ratio 1:30, and treated with 0.5M $NaHCO_3$.

Detection of uranium in soil

A method of Solid State Nuclear Track Detectors ($SSNTD_s$) was used in the detection of depleted uranium in soil sample.

- The Track Detectors

Commercially available sheets of CR-39 plastic which are presently known to be the most sensitive SSNTDs and also characterized by low background were used in the present work.

These detector sheets of 250 µm thick where cut into small pieces each of (1 cm x 1 cm) area. The present sheets of CR-39 were made by Pershore modeling limited Company, U.K. The detector sheets were stored at normal laboratory conditions.

- Experimental Procedure for uranium concentration measurement in soil

The soil samples were prepared as previous suggestion.0.5g of soil samples were mixed with 0.1 g of methylcellulose powder ($C_6H_{10}O_5$) used as a binding material. The mixture was pressed by using a mechanical compressing device with force equal to (5 tons) in to a pellet of 1 cm diameter and 1.5mm thickness.

The pellets were covered with (CR-39) detector and placed in a plate of paraffin wax at a distance of (5 cm) from the neutron source (Am-Be), with flounce of thermal neutron (3.024 x10^{9} n.cm^{-2}) and flux (5 x 1 0^{3} n. cm^{-2}.s^{-1}), to obtain induced fission fragments from the

$$^{235}\mathrm{U} + {}^{1}_{0}\mathrm{n}_{\mathrm{thermal}} \rightarrow {}^{236}\mathrm{U} \rightarrow {}^{92}_{36}\mathrm{Kr} + {}^{141}_{56}\mathrm{Ba} + 3\mathrm{n} + \mathrm{Q} + 0.85\ \mathrm{Mev} \qquad (5)$$

After the irradiation time (7 days) , (CR-39) detectors were etched in (6.25N) NaOH solution at temperature of 60 °C for (6 h) , then the induced fission tracks density were recorded using the optical microscope.

The metal's samples were cut in small pieces and irradiated as mentioned in the above procedure for soil.

The density of fission tracks (ρ) in the samples was calculated according to the following relation [17].

Track detectors (ρ)=Average number of total pits(tracks)/Area of field view.

The uranium concentrations in soil samples were measured by comparison between track densities registed on the detectors of the sample pellet and that of the standard geological sample pellets from the relation [18,19]**(Fig.(1)):**

$$Cx(\text{sample})/\ \rho x(\text{sample})=Cs(\text{standard})/\ \rho s(\text{standard}) \qquad (6)$$

$$Cx=Cs.(\ \rho x/\ \rho s).$$

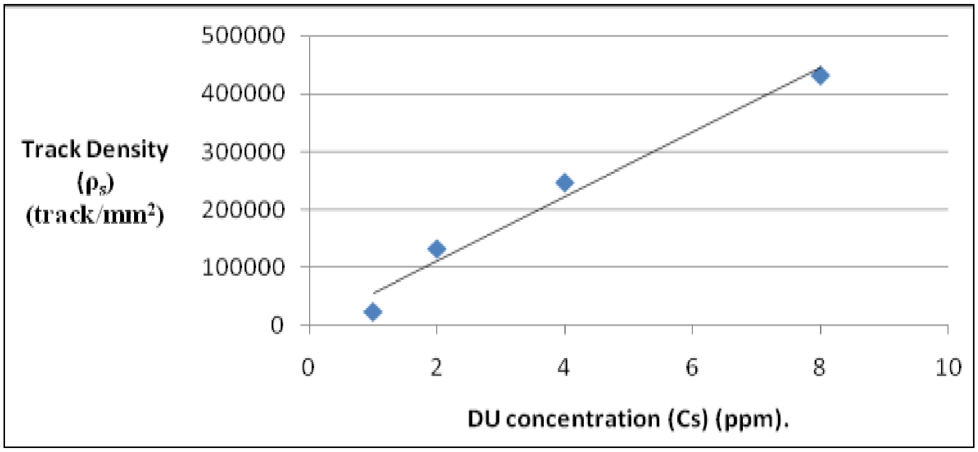

Fig.(1):Relation between standard track density and standard DU concentration[19]

Where:

Cx: uranium concentration in unknown sample (ppm).

Cs: uranium concentration in standard sample (ppm).

ρx: track density of unknown sample (track/mm^2).

ρs: track density of standard sample (track/mm^2).

RESULTS & DISCUSSIONS

Time Effect

The removal rate of uranium at various leaching time (2, 3, 4, 5 and 7days) was studied and it was found that the removal rate increased with increasing time, the concentrations of uranium after treatment were obtained between [6.903 ppm (2 days) to 4.54 ppm (7 days) with best removal efficiency equal to (63.73%) at (14 ^{0}C).

The concentration of DU after treatment were decreased gradually because the soil particles exposed more to solution, and that gave the chance to all the DU particles to reacts with the solution, the last two samples were approximately equal in DU removal, this was because the solution reached the saturation level and no important decrease were regarded. Relation between time and DU concentration plotted in Fig. (2).

Temperature Effect

Five soil samples with five values of temperature (20, 30, 40, 50 and 60) ^{0}C gave uranium concentration [from 5.857 ppm (20 ^{0}C) to 4.483 ppm (60 ^{0}C)] with best removal efficiency of DU=64.2%

It was regarded that the uranium concentrations obtained from the treatment were decreased with increasing temperature.

Temperature more than 60 ^{0}C was not taken, because $NaHCO_3$ will disintegrate to its ions in this temperature degree [20]. Relation between temperature and uranium concentration were plotted in Fig(3).

CARBONATE CONCENTRATION EFFECT

Five concentrations of $NaHCO_3$ (0.2, 0.3, 0.5, 0.7, and 0.8M) were examined. The resulted uranium concentration were decreased slightly with increasing sodium bicarbonate molarity [4.924 ppm at (0.2M) to 4.328 ppm at (0.8M)], the best removal efficiency of DU were equal to (65.4%).

All the previous sets and this set , the solution color after treatment was (light yellow), but for (0.8M)was different ,it had a green color, this color might due to the oxides of uranium which had different colors, and the green color referred to U_3O_8[21], the yellow color referred to UO_3 oxide. The relation between carbonate molarity and uranium concentration was plotted in fig(4).

CARBONATE/BICARBONATE RATIO EFFECT

Three samples were prepared with three different ratios of Na_2CO_3 to $NaHCO_3$, two similar results were obtained for the (S1c1/b) and (S1c/b1), the two samples had either carbonate or bicarbonate to be the dominant, and gave the same effect in treatment (about 4.6 ppm), but the sample S1c/b had equal weight of (HCO_3^{-1}) and (CO_3^{-2}), so it gave a great effect in treatment of (1.47 ppm of DU) with removal efficiency (88.25%), this difference in the treatment from S1c/b and the other two samples was due to the high concentration of (HCO_3^{-1}) and/or (CO_3^{-2}) which prevented the precipitation of uranium as hydroxide in the solution[7]. Relation between carbonate/bicarbonate and DU concentration was plotted in fig.(5). It can observe from this figure that the optimum value for Carbonate/Bicarbonate ratio equal to about 1.25 which give about 1.1ppm of Depleted Uranium. The three solutions of the samples colored with light green after treatment, and this might be resulted from the existence of (U_3O_8) in sample solution.

PH EFFECT

The effect of pH on the DU removal efficiency from soil by leaching showed in Fig. (6). (the uranium concentration versus pH values), Solutions' pH were adjusting by pH meter. The uranium removal efficiency decreased with increasing pH solution, the best treatment were in pH = 6.53(5.235 ppm of DU), with best removal efficiency of (58.2%). Where the oxidized uranium (VI) is soluble at low pH[5].

The treatment efficiency of samples set were not differed greatly, sample of (pH=9.1) had a very light green color solution after treatment, while a sample of (pH=6.53) had a yellow color.

OXIDATIVE REAGENT EFFECT

Four samples in this set were prepared; one of the samples was prepared to show the treatment efficiency without oxidation and in the same experiment conditions the other three samples were prepared (with the use of $KMnO_4$) to show the effect of oxidation on the treatment process.

The best treatment was in S1O1 (4.95 ppm) with removal efficiency of (60.4%) at (0.2gm ($KMnO_4$)/1 gm of soil) and with further increasing of $KMnO_4$ concentration the removal efficiency were decreased, this was happened because the increase of $KMnO_4$ in the solution decrease the $KMnO_4$ capacity to solubilize the DU, and some of $KMnO_4$ were not reacted and still in the solution.

This was clearly regarded when the slight violet color appeared in the solution of S1O2, S1O3, but in S1O1 the violet color disappeared completely and the oxide gave the best treatment result in this set with the completely reaction of $KMnO_4$ in solution.
Fig.(7).represented the relation between oxide and DU concentration.

SOIL /SOLUTION RATIO EFFECT

In this set five different soil/solution ratio were used. The uranium concentration in soil after treatment were widely differed from first sample and the last one (8.086ppm to 4.87ppm) and the best removal efficiency equal to (61.08%), where the concentration of DU were decreased with increasing soil/solution ratio.

This was happened because those DU particles have a great chance to react and solubilize with a high ratio of solution.

Not important notes about the solution were recorded; it had the same ordinary color of (light yellow). Relation between DU concentration and ratios were plotted in fig.(8).

RINSING EFFECT

The first sample in this set were prepared before rinsing and the two other were after rinsing, after one day and two days respectively, the best one were after two days (4.3 ppm) with removal of (65.6%).Fig(9).displayed the rinsing effect.

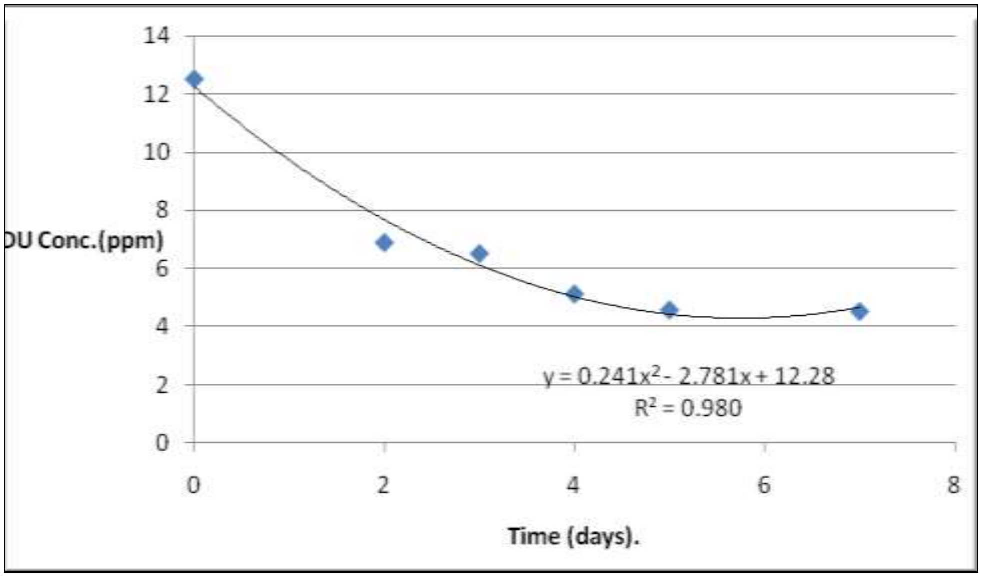

Fig.(2):Relation between DU concentration & time of leaching

(Temp.=14 ^0C , pH=8.06 ,Solution Conc.=0.5M $NaHCO_3$,Soil:Solution=1:20).

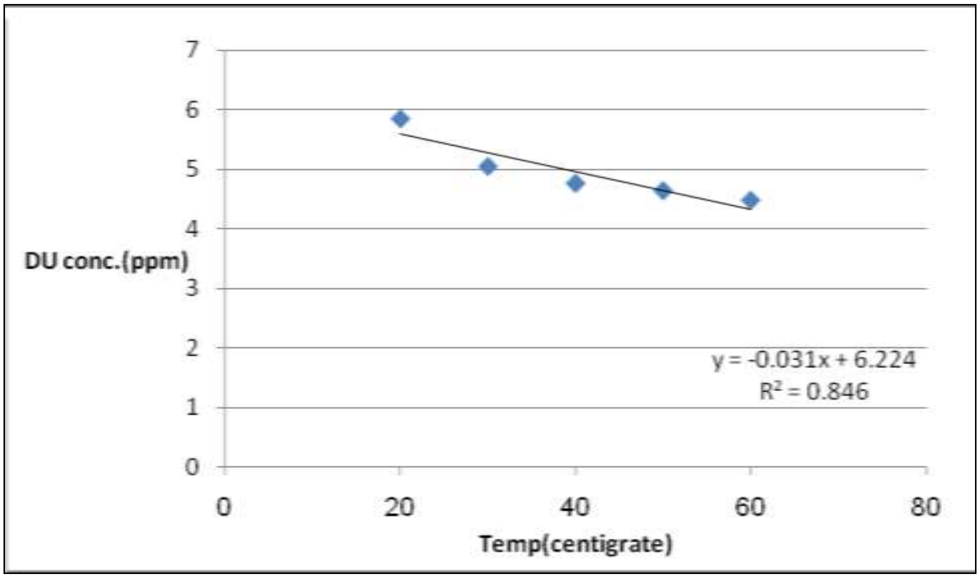

Fig.(3):Relation between DU concentration & solution's temperature.

(Leaching time=3 and half hour , pH=8.06 , Solution Conc.=0.5M $NaHCO_3$,Soil:Solution=1:20).

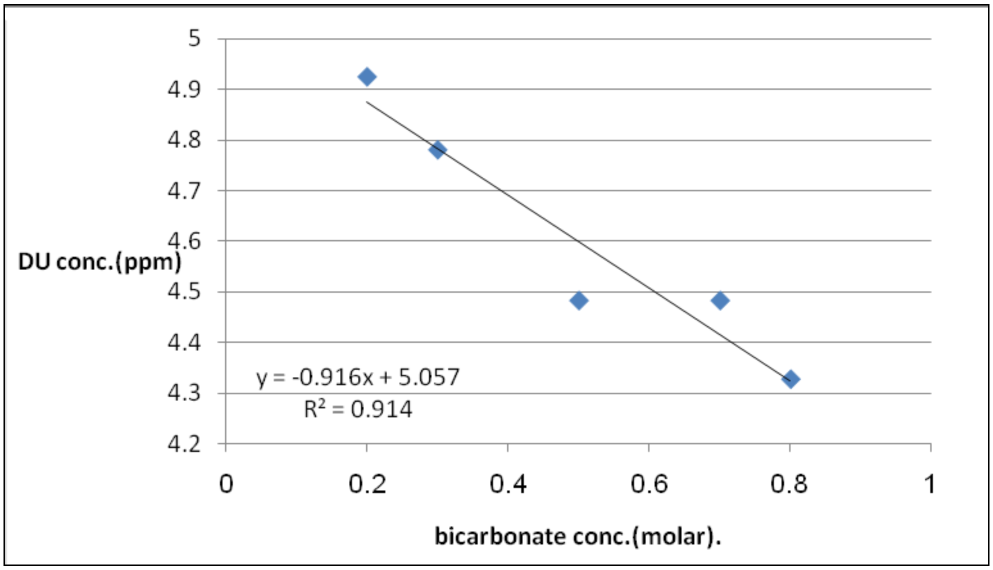

Fig.(4):Relation between DU concentration & $NaHCO_3$ concentration

(Temp.=23 ^0C , pH=8.06 ,Leaching time=4 days ,Soil:Solution=1:20).

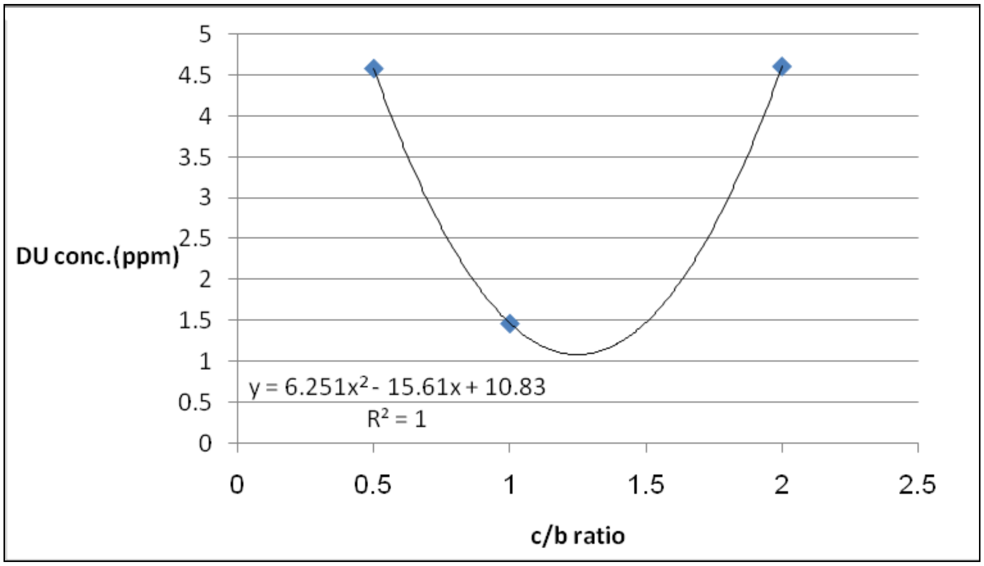

Fig.(5):Relation between DU concentration & $NaHCO_3/Na_2CO_3$ Ratio.

(Temp.=26 ^0C,Leaching time=3 days , Soil: Solution=1:30).

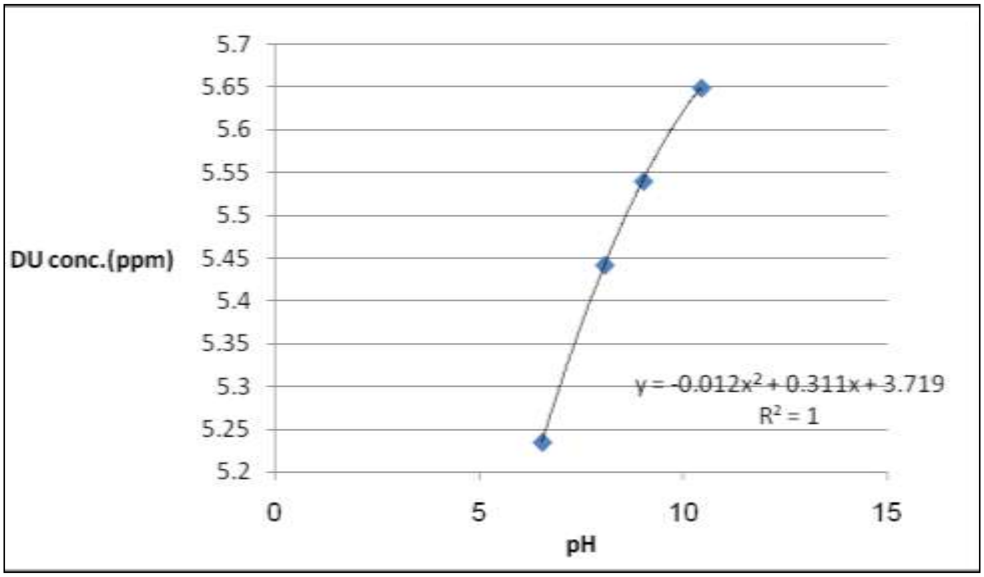

Fig.(6):Relation between DU concentration &PH of solution. (Temp.=31 °C,Leaching time=2 days , Solution Conc.=0.5M $NaHCO_3$,Soil:Solution=1:30).

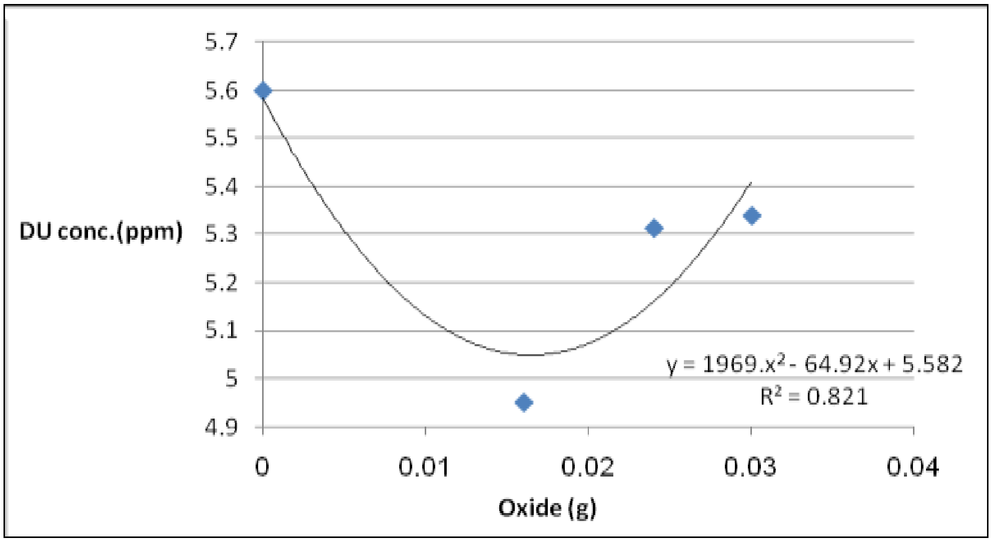

Fig.(7):Relation between DU concentration & Oxide's concentration. (Temp.=25 °C,Leaching time=2 days , solution conc.=0.5M $NaHCO_3$,soil:solution=1:30).

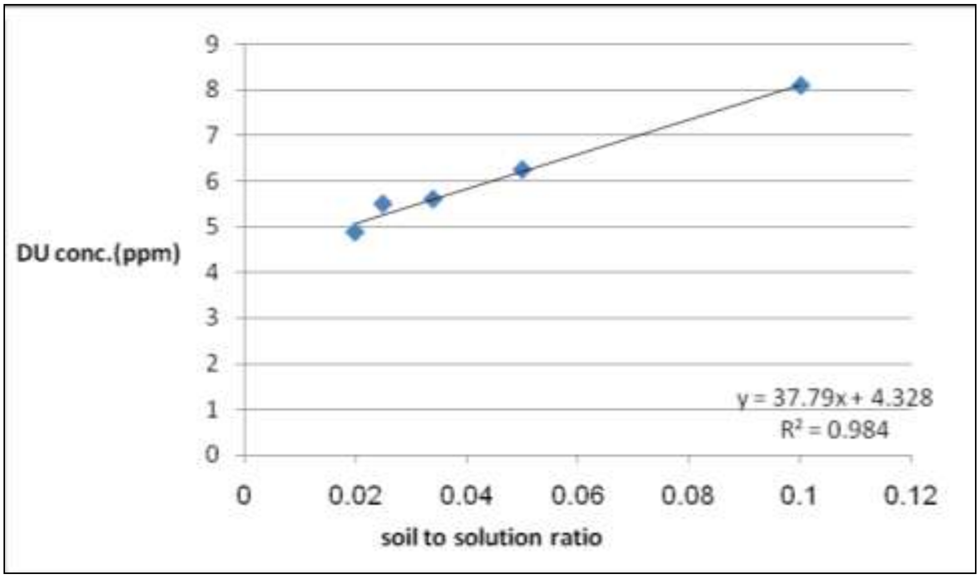

Fig.(8):Relation between DU concentration & soil to solution ratio.

(Temp.=19 °C,Leaching time=2 days , Solution Conc.=0.5M $NaHCO_3$).

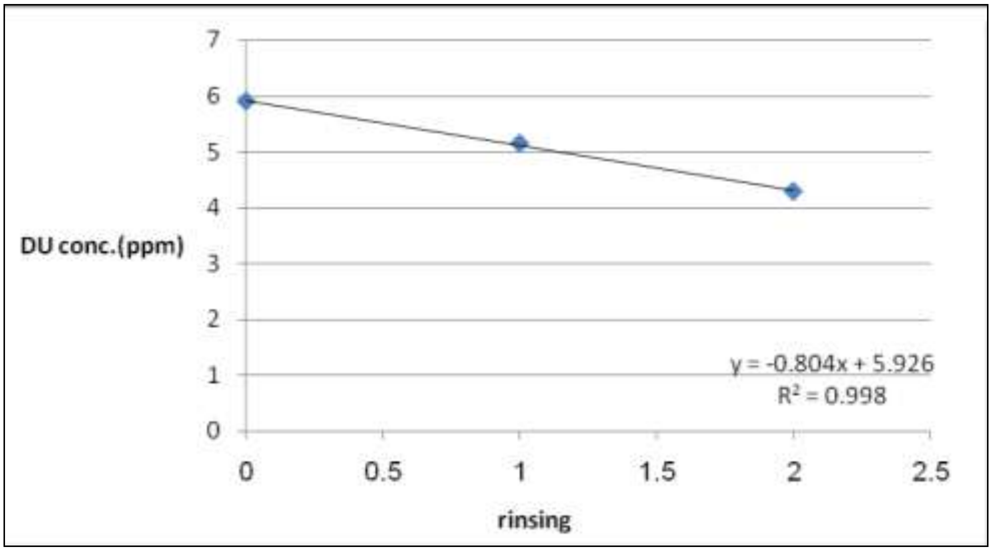

Fig.(9):Relation between DU concentration & Rinsing after treatment.

(Temp.=26 °C,Leaching time with 0.5M $NaHCO_3$ =2 days, Soil:Solution=1:30).

Conclusions

a. with increasing leaching time the DU concentration in soil after treatment were decreased. Removal efficiency were ranged between (44.8-63.7)%

b. With increasing solution temperature the DU concentration after treatment in soil were decreased. Removal efficiency were ranged between (53.2-64.2)%

c. With increasing Bicarbonate concentration, the DU concentration in soil after treatment were decreased. Removal efficiency were ranged between (60.6-65.4)%

d. Carbonate/bicarbonate ratio: the best treatment was at ratio=1, when the weight of carbonate were equal to bicarbonate. Removal efficiency were ranged between (63.1-88.25)%.that was good result to decrease the DU concentration lower than the allowed level of 40 Bq/Kg or about 1.6 ppm.

e. With increasing pH the DU concentration in soil were also increased. Removal efficiency were ranged between (60.8-54.8)%

f. The influence of oxidizing reagent were decreased as the increasing of its weight added to solution, the best treatment were at oxidizing ratio to soil(0.02g $KMnO_4$/1g of soil). Removal efficiency were increased from (2.3 to 5.2)% from before oxidation.

g. With increasing the soil/solution ratio the DU concentration in soil were decreased. Removal efficiency were ranged between (35.4 - 61.08)%

h. Taking two conditions of rinsing with distilled water giving good effect after treatment with bicarbonate leaching. Removal efficiency increased from (6 to12.8) % from before rinsing.

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Sets	Sample name	Description.
Time set	S1a	Sample 1 at time=2 days
	S1b	Sample 1 at time=3 days
	S1c	Sample 1 at time=4 days
	S1d	Sample 1 at time=5 days
	S1e	Sample 1 at time=7 days
Temperature set	S1t1	Sample 1 at temp.=20 C^o
	S1t2	Sample 1at temp.=30 C^o
	S1t3	Sample 1at temp.=40 C^o
	S1t4	Sample 1at temp.=50 C^o
	S1t5	Sample 1at temp.=60 C^o
$NaHCO_3$ concentration set	S1k1	Sample 1 at M=0.2
	S1k2	Sample 1 at M=0.3
	S1k3	Sample 1 at M=0.5
	S1k4	Sample 1 at M=0.7
	S1k5	Sample 1 at M=0.8
Carbonate/Bicarbonate Ratio set	S1c1/b	Sample 1at $Na2CO_3 > NaHCO_3$
	S1c/b1	Sample 1at $Na2CO_3 < NaHCO_3$
	S1c/b	Sample 1at $Na2CO_3 = NaHCO_3$
pH set	S1pH1	Sample 1at pH=6.53
	S1pH2	Sample 1at pH=9.01
	S1pH3	Sample 1at pH=8.06
	S1pH4	Sample 1at pH=10.43
Oxidative reagent set	S1bo	Sample 1 before oxidation
	S1o1	Sample 1 at KMnO4=0.02/1g soil
	S1o2	Sample 1 at KMnO4=0.03/1g soil
	S1o3	Sample 1 at KMnO4=0.04/1g soil
Soil/Solution ratio	S1r1	Sample 1at soil/solution ratio=1:10
	S1r2	Sample 1at soil/solution ratio=1:20
	S1r3	Sample 1at soil/solution ratio=1:30
	S1r4	Sample 1at soil/solution ratio=1:40
	S1r5	Sample 1at soil/solution ratio=1:50
Rinsing Effect	S1br	Sample 1before rinsing
	S1rr1	Sample 1 after one rinsing
	S1rr2	Sample 1 after two rinsing
Other Solution	S1ca	Sample 1 with citric acid
	S1sa	Sample 1 with sulfuric acid

	S1ci/bi	Sample 1with citric acid then $NaHCO_3$