Extraction of Uranium and Thorium Using Triphenylphosphine Oxide (TPPO) From El Sela Granite, South Eastern Desert, Egypt

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Abstract: Triphenylphosphine Oxide (TPPO) dissolved in toluene has been chosen for extraction both of uranium and thorium. Different parameters were carried out testing the following variables: different diluents, TPPO concentrations, aqueous to organic phase ratio (A/O), shaking time, temperature and different stripping agents. The thermodynamic parameters \( \Delta H \), \( \Delta G \) and \( \Delta S \) were calculated for uranium and thorium extractions. The data obtained showed that, best results for uranium were obtained using each of the following systems; 0.02M TPPO dissolved in toluene, A/O 4:1, 5 min. of shaking time at room temperature and 0.5M HNO\(_3\) for stripping of uranium. On other hand, 0.04M TPPO dissolved in toluene, A/O 4:1, 4 min. of shaking time at room temperature and 2M HNO\(_3\) acid was used for thorium stripping. The factors studied were followed by application of the extraction system to determine the uranium and thorium from studied samples, El Sela area, south Eastern Desert, Egypt. Thermodynamic were identified proving exothermic, spontaneous and randomness reaction for extraction processes.

Keywords: Uranium, Thorium, Extraction, TPPO, Toluene

1. Introduction

Gabal El Sela lies between longitude 36° 14′ 08.78″ E and latitude 22° 17′ 55.83″ N, covering an area about 70 Km\(^2\) of basement rocks (Fig.1). El Sela granite was actually considered as highly weathered uraniferous granite and subjected to severe alteration processes in southern Egypt and was displayed high uranium content, Abdel Meguid et al. [1].

The area of study is located in the southern Eastern Desert of Egypt between Lat. 22° 14′ 30″ and 22° 18′ 36″ N and Long.36° 11′ 45″ and 36°16′ 30″ E. The country rock (El Sela granite) is composed mainly of pink to pinkish grey coarse grained biotite granite and fine grained red to pink two mica granites. These granites are cavernous, uraniferous and extremely weathered in arid to semi-arid climatic conditions. The area was subjected to successive tectonic events followed by magmatic intrusions especially in the sheared zone at the northern part. Some of these intrusions were enriched in uranium and mostly associated with high fluid phases evidenced by the presence of different alteration haloes, Abdel Meguid et al. [1]; Ibrahim et al. [2]; Abd El Naby and Dawood [3].

Abdel Meguid et al.[1], indicated that El Sela granite displays high uranium content in the form of primary and secondary uranium minerals. They specified uraninite, pitchblende, coffenite, autunite and uranophane minerals as evident source for uranium intragranitic deposit. These U-minerals are enriched in the shear zone cross cut in the northern studied area.

This shear zone represent deep, long and wide paleo-channels for the magmatic activities which were associated with high fluid phases as indicated by numerous alteration features and high weathering intensity that affected El Sela granite.

Ibrahim et al. [4], stated that the geological, mineralogical and the geochemical criteria of the studied area as well as the prevailing of arid to semi-arid weather conditions have been favorable for forming a pedogenic surficial uranium deposit in the northern part of the area.

Ibrahim et al. [2], have studied in detail the U-potentiality and recovery from El Sela ore material. The applied uraniferous Sela sample experiment assayed 950 U ppm and recovered in the form of sodium di-uranate (Na\(_2\)U\(_2\)O\(_7\)). They found that El Sela uranium ore is easily leachable.

Ibrahim et al. [5], have applied leaching process on technological scale sample for studying the mining ability, leaching characteristics and the recovering conditions, they found that El Sela U-ore material is easily mineable and easily recovering.

Mira and Ibrahim [6], evidenced the biogenic origin of the tetravalent uranium minerals where they recorded natural interaction between the uranium and the bacteria and fungi species within the studied rock.
Gamil et al. [7], applied a hydrometallurgical process to extract U and Th from El Sela waste sample. Chemical analysis of the produced leach liquor revealed that; it assays 390 Uppm and 120 Thppm with leaching efficiencies reached 78% and 75% respectively. This result assured that El Sela U-ore material still in needs to further experimental leaching techniques to get the optimum recovery percent.

The uranium minerals in El Sela samples were accumulated through the successive hydrothermal and meteoric fluids events (Fig.2). El Sela area reveals the presence of other economic minerals such as rutile, zircon, columbite, samarskite, monazite and thorite. Some uranium anomalous sites along the NW-SE gashes were recorded and chosen for manual trenching to check the radioactive minerals and their genesis, Ibrahim et al. [8]. Some of these trenches, T1, T2, T3, T4 and T5 have been chosen to develop the excavation work as box-cuts (Fig.3).

The present work will concern by extraction of uranium and thorium to reach the optimum extraction efficiency from El Sela uranium ore. To achieve this target, three representative samples from El Sela uranium ore materials were taken from the box cut No. T5 on three different depths (1.5, 2.5 and 3.5 m). The uranium spectrometric measurements within this box-cut indicate as shown in (Fig. 4).

Uranium and thorium are used extensively in nuclear energy programs; hence several methods were required for its separation and determination. Solvent extraction is one of the most common and important methods for separation of many elements and always proved itself very helpful as a recovery method for many components. Uranium (VI) was extracted using different organic extractants such as trioctylphosphine oxide (TOPO) dissolved in cyclohexane, Horton and White [9]. Di (2-ethylhexyl) phosphoric acid (DEHPA) diluted in carbon tetrachloride (CCL) was used for extraction of U (VI) and determined by infrared spectroscopy, Kiwan and Amin [10]. Whereas tri (2-ethylhexyl) phosphate (TEHP) was used for extraction of U (VI), Sundaramrthi et al. [11]. Uranium was extracted using tributylphosphine oxide (TBPO) diluted.
in toluene and determined by fluoride fusion method, Yasumasa and Hideaki [12].

Many solvents containing amine group were applied to extract uranium as 8-hydroxyquinoline, Isea et al. [13]. Meanwhile, uranium was recovered using cyanex-923, Gupta et al. [14]. Selective extraction and separation of uranium and thorium was carried out using N-phenylbenzeno-18-crown-6-hydroxamic acid (PBCHA), Agrawal and Vora [15]; Agrawal et al. [16].

Extraction of uranium (VI) by di-2,4,4-trimethyl-bentyl phosphoric acid (cyanex – 272) dissolved in toluene, Madane et al. [17]. While, tributyl phosphate (TBP) was used for extraction of U (VI), McKay and Streeton [18]. Uranium (VI) and thorium (IV) were extracted from phosphate medium using a synergistic DOPPA–TOPO mixture and determined spectrophotometric method, Krea and Khalaf [19].

Cyanex 272, Cyanex 302 and TBP were used for extraction of thorium (IV) from 0.5 M nitric acid, Nasab et al. [20]. XAD-4 was utilized for selective separation, preconcentration and determination of lanthanum (III), cerium (III), thorium (IV), uranium (VI) and determined by (ICP-AES), Jain et al.[21]. Uranium and thorium were separated from nitrate medium using Amberlite CG-400 and determined using ICP-MS, Rozmari et al. [22]; Akkaya [23].

2. Experimental

2.1. Instrumentations

Double distilled water was used for preparing all standard solutions and reagents using Aquatron 4L/h (England). The hydrogen ion concentration for the solution was measured using Inolab digital pH-meter, level 1 (England), with an error of ±0.01 at ambient laboratory temperature. The reagents used in this work were weighed using an electronic analytical balance Shimadzu AY 220 (Germany) giving a maximum sensitivity of 10⁻⁶ g and an accuracy of ±0.01%.

The analysis of SiO₂, Al₂O₃, P₂O₅, TiO₂, uranium and thorium were determined by spectrophotometer Metrotech Inc model Sp-8001, (Germany) with the range 200-1100 nm with an accuracy of ±1nm. One match of 5 cm² quartz cell with a pass length of 1cm was used for both samples and blank reagent. The analysis of calcium, magnesium and iron were determined by titration, Shapiro and Brannock [24]. Sodium and potassium were determined by a Sherwood flame photometer model 410 (England), using a series of chemical standard solutions.

2.2. Chemicals used

All chemicals and reagents used were of analytical grade (AR). Uranium stock solution was prepared by dissolving 2.109 g of UO₂ (NO₃)₃·6H₂O with purity > 99.99 % in a definite volume 1000 ml double distilled water containing 2 ml of 6M HNO₃ acid to get a fixed concentration of 1000 ppm. The different concentrations of uranium were prepared by dilution.

While, thorium stock solution were prepared by dissolving 2.38g of Th(NO₃)₄·4H₂O dissolving in 1000 ml double distilled water containing 1ml concentrated nitric acid. Uranium and thorium were also measured by Arsenazo-III, Nenodruck and Glukhova [25]; Borak et al.[26]; Marczenko [27].

In the present work, several attempts have been made to extract and determine uranium and thorium using triphenylphosphine oxide (TPPO) as organic phosphorus compound from nitric acid medium with chemical formula of C₉H₆P₂O and a molecular weight of 278.28 g/mol.

Reviewing the different leaching and extraction methods of uranium and thorium from El Sela ore material, approve the success of TPPO as extractant and highly selectivity for uranium and thorium compared to other processes for extraction. The present work will concern by applying triphenylphosphine oxide (TPPO) to extract uranium and thorium from El Sela uranium ore material.

3. Results and Discussion

The extraction of uranium (VI) and thorium (IV) were carried out by applying the batch technique. The triphenylphosphine oxide (TPPO) was prepared by dissolving different concentrations in different diluents to prepare the organic phase. This organic phase was added to the uranium or thorium solution in a separating funnel and shaken well according to certain contact time. After equilibration, a complete separation of the two phases was carried out. The uranium or thorium measured spectrophotometrically in the aqueous phase by the Arsenazo III method.

3.1. Factors affecting the extraction of uranium (VI) and thorium (IV).

Several experiments were done to study factors controlling the extraction of uranium and thorium such as diluent types, extractant concentrations, aqueous to organic (A/O) phase ratio, shaking time, temperature, thermodynamic and different stripping agents.

Different concentrations from 0.02 to 0.4 M of triphenyl phosphate oxide (TPPO) were prepared in appropriate diluents such as toluene (C₆H₅CH₃), kerosene (C₁₂H₂₅), benzene (C₆H₆), cyclohexane (C₆H₁₂) or chloroform (CHCl₃).

3.1.1. Effect of diluents

To choose which diluent is the best for uranium or thorium extraction, (TPPO) was dissolved in different diluents namely: toluene, benzene, kerosene, chloroform, and cyclohexane. This organic phase was shaken to the aqueous phase containing uranium or thorium solution at the ambient room temperature (22 ± 1°C).

From the experiments applied for checking the suitable diluents, it was found that, TPPO in toluene as a diluent gives high extraction efficiency, (98.5 %) to extract uranium from solution if compared with benzene (92 %), cyclohexane (89 %), kerosene (85 %) and chloroform (80 %) for one contact.
It means that, the priority at which the extraction increases is toluene > benzene > cyclohexane > kerosene > chloroform (Fig.5).

On other hand, it was found that TPPO in toluene as a diluent gives high extraction efficiency, (97.5 %) to extract thorium from solution if compared with benzene (90 %), cyclohexane (80 %), kerosene (75 %) and chloroform (55 %) for one contact. It means that, the priority at which the extraction increases is toluene > benzene > cyclohexane > kerosene > chloroform (Fig.5).

50 60 70 80 90 100
Toluene Benzene Cyclohexane Kerosene Chloroform
Diluents
% E
U Th
98.5 %
97.5 %

Extraction conditions for uranium: Uranium concentration: 100 ppm, 0.04 M TPPO used as extractant dissolved in different diluents, A/O:2/1, at ambient room temperature and shaking time:5min.

Extraction conditions for thorium: Thorium concentration: 100 ppm, 0.04 M TPPO used as extractant dissolved in different diluents, A/O:2/1, at ambient room temperature and shaking time:5min.

Figure 5: Effect of different diluents on uranium and thorium extraction (%E).

3.1.2. Effect of Extractant Concentrations
Different concentrations from 0.02 to 0.4 M of (TPPO) in toluene had been applied during the experiments run to study the extraction percent (%E) of uranium or thorium. By changing the extractant concentrations of TPPO from 0.02 to 0.4 M, it was found that the %E of uranium reach to its maximum extraction percent (99 %) at 0.02 M of TPPO in toluene. While, extraction of thorium was (97.5 %) at 0.04 M (Fig.6).

Extraction conditions for uranium: Uranium concentration: 100 ppm, TPPO dissolved in toluene, A/O:2/1, at ambient room temperature and shaking time:5min.

Extraction conditions for thorium: Thorium concentration: 100 ppm, TPPO dissolved in toluene, A/O:2/1, at ambient room temperature and shaking time:5min.

Figure 6: Effect of extractant concentrations on uranium and thorium extraction (%E).

3.1.3. Effect of aqueous to organic phase ratio (A/O)
The effect of aqueous phase (A) to organic phase (O) ratio on the extraction of uranium and thorium were studied covering the range 1:2, 1:1, 2:1 and 4:1 while the other factors were kept constant. The obtained results indicated that the ratio 4:1 showed the best extraction percentage (98.5 %) for uranium and (97.5 %) for thorium (Fig.7). This is due to steric effect resulting from the presence of three bulky phenyl groups leading to slow the reaction markedly. According to this phenomenon, it was found that the TPPO is more efficient in uranium or thorium extraction in case of high aqueous percent (%).

50 60 70 80 90 100
1:2 1:1 2:1 4:1
A/O phase ratio
% E
U Th
98.5 %
97.5 %

Extraction conditions for uranium: Uranium concentration: 100 ppm, TPPO dissolved in toluene, Extractant conc. 0.02 M TPPO, at ambient room temperature and shaking time: 5 min.

Extraction conditions for thorium: Thorium concentration: 100 ppm, TPPO dissolved in toluene, Extractant conc. 0.04 M TPPO, at ambient room temperature and shaking time: 5 min.

Figure 7: Effect of A/O phase ratio on uranium and thorium extraction (%E).

3.1.4. Effect of Shaking Time
The effect of shaking time on the extraction of U (VI) or Th (IV) were studied by varying the shaking time from 0.5 to 10 minutes using (TPPO) as extractant. It was found that, the % E of uranium changes from 90 % with 0.5 min. to 98.5 % with 5 min. shaking time, then the % E were kept constant till 10 minutes. Therefore, the required shaking time to extract most uranium (VI) from its solution was chosen as 5 minutes (Fig.8).

On other hand, the % E of thorium changes from 88 % with 0.5 min. shaking time to 97.5 % with 4 min., then the % E were kept constant till 10 minutes. So that, the required shaking time to extract most Th (IV) was chosen as 4 minutes (Fig.8).
3.1.5. Effect of Temperature
The effect of temperature on the extraction of U (VI) or Th (IV) was studied by varying the temperature from ambient room temperature (22 ± 1 ºC) to 100º C. It was found that the % E of uranium changes from 98.5 % with 22º C to 50 % with 100º C. So, the required temperature to extract most U (VI) was chosen at ambient room temperature, (22 ± 1 ºC). While, the % E of thorium ranges from 97.5 % with 22º C to 42 % with 100º C. So, the required temperature to extract most Th (IV) was 22º C (Fig.9).

3.1.6. Effect of thermodynamic for uranium and thorium extraction
Enthalpy (ΔH) is a defined thermodynamic potential or expression of system energy changes. The enthalpy is the heat absorbed or released by the material through a chemical reaction. The change ΔH is positive, the reaction is heat releasing and exothermic processes, while ΔH is negative, the reaction is heat releasing and exothermic processes. While, ΔG is negative, the reaction is heat releasing and exothermic processes, Moran and Shapiro [28].

Free energy (ΔG) is the reaction spontaneous or non spontaneous through the chemical reaction. The change ΔG is positive, the reaction is non spontaneous and it needs temperatures, pressures and catalysis. While, ΔG is negative, the reaction is spontaneous and it doesn't require temperatures, pressures and catalysis, Walter et al. [29]; Brachman [30].

Entropy (ΔS) means that, the element or ion moved and transferred to the other phase in any site or any place without need and require the arranged, number of moles and quantities. The change ΔS is positive, this means increase in randomness. While, ΔS is negative this means decrease in randomness, Mark [31]; Sandler [32]; Ben [33].

The extraction of metal complex into an organic phase involves large changes in enthalpy ΔH (salvation processes) and entropy ΔS (solvent orientation and restructuring), leading to considerable temperature effects.

The results are shown in (Table 1) and Figs. (10 and 11) as a function of Log D versus 1000/T, K⁻¹ give a straight line whose slope equals (-ΔH / 2.303R) for the extraction of U (VI) and Th (IV) by TPPO in toluene from nitrate medium.

## Table 1: Effect of temperatures (ºK) on uranium and thorium distribution coefficient

<table>
<thead>
<tr>
<th>Temp., (ºC)</th>
<th>Temp., (ºK)</th>
<th>1000/T, K⁻¹</th>
<th>Log D (U)</th>
<th>Log D (Th)</th>
</tr>
</thead>
<tbody>
<tr>
<td>22</td>
<td>295</td>
<td>3.38</td>
<td>2.42</td>
<td>2.192</td>
</tr>
<tr>
<td>25</td>
<td>298</td>
<td>3.36</td>
<td>2.24</td>
<td>1.958</td>
</tr>
<tr>
<td>30</td>
<td>303</td>
<td>3.3</td>
<td>2.07</td>
<td>1.725</td>
</tr>
<tr>
<td>35</td>
<td>308</td>
<td>3.24</td>
<td>1.810</td>
<td>1.576</td>
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<tr>
<td>40</td>
<td>313</td>
<td>3.19</td>
<td>1.55</td>
<td>1.427</td>
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<tr>
<td>45</td>
<td>318</td>
<td>3.14</td>
<td>1.39</td>
<td>1.315</td>
</tr>
<tr>
<td>50</td>
<td>323</td>
<td>3.09</td>
<td>1.23</td>
<td>1.204</td>
</tr>
<tr>
<td>55</td>
<td>328</td>
<td>3.04</td>
<td>1.155</td>
<td>1.107</td>
</tr>
<tr>
<td>60</td>
<td>333</td>
<td>3</td>
<td>1.079</td>
<td>1.011</td>
</tr>
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</table>

**Figure 10: A plot of log D against 1000/T for uranium (VI) extraction.**
Figure 11: A plot of log D against 1000/T for thorium (IV) extraction.

From (Table 2), the ∆H value for U (VI) was (-70.27 kJ/mol) and for Th (IV) was (-55.97 kJ/mol) as calculated from the slope using the Van’t Hoff equation Eq. (1).

\[
\log D = -\frac{\Delta H}{2.303 RT} + C
\]  
(1)

Where D is the distribution coefficient, ∆H the enthalpy change for the extraction reaction, R is the universal gas constant (8.314 J/mol k) and C is an integration constant.

The negative value of ∆H indicates that the extraction of U (VI) and Th (IV) in this system is an exothermic process. The negative value of (ΔS) suggests the decreased randomness at TPPO/ uranium or thorium interface during the extraction processes. This randomness state is mostly due to the extraction of uranium (VI) or thorium (IV) into active sites of (TPPO) to form stable structure. On other hand, the negative value of ∆G indicates that the extraction reaction is spontaneous.

3.1.7 Effect of different stripping agents.

Different stripping agents were tried for back extraction of U (VI) or Th (IV) from the organic phase, the stripping was tested using certain stripping agents such as nitric, sulphuric, ortho-phosphoric and hydrochloric acid. The acids molarity ranged from 0.5 to 6M.

It was found that, 97.5, 90, 75 and 65 % U can be stripped by 0.5M HNO₃, 1M H₂SO₄, 0.5M H₃PO₄ and 0.5M HCl acid, respectively. Accordingly, 0.5M HNO₃ acid was chosen as the best stripping agent for uranium (Fig.12).

While, 96, 89, 72 and 68 % Th can be stripped by 2M HNO₃, 0.5M H₂SO₄, 0.5M H₃PO₄ and 0.5M HCl acid, respectively. Accordingly, 2M HNO₃ acid was chosen as the best stripping agent for thorium (Fig.13).

Table 2: Thermodynamic parameters for the uranium and thorium extractions

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>Temp. (°K)</th>
<th>∆H, (kJ/mol)</th>
<th>∆G, (kJ/mol)</th>
<th>∆S, (J/mole, K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>U</td>
<td>Th</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
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<td>60</td>
<td>333</td>
<td>-6.88</td>
<td>-2.60</td>
<td>-190.36</td>
</tr>
</tbody>
</table>

Extraction conditions for uranium: Uranium concentration: 100 ppm, TPPO dissolved in toluene. Extractant conc. 0.02 M TPPO, A/O :4/1, at ambient room temperature and shaking time 5min.

Figure 12: Effect of different stripping agents on stripping process of U from TPPO.

Extraction conditions for thorium: Thorium concentration: 100 ppm, TPPO dissolved in toluene, Extractant conc. 0.04 M TPPO, A/O :4/1, ambient room temperature and shaking time 4min.

Figure 13: Effect of different stripping agents on stripping process of Th from TPPO.

Application on the studied samples from El Sela area, south Eastern Desert, Egypt

The studied samples were taken from T5 trench in El Sela shear zone on different depths (1.5m, 2.5m and 3.5m). All these samples are altered products of granite (kaolinitized granite).

The studied samples were completely attacked using mixture of acids HF, HNO₃, HClO₄ and HCl. Shapiro and Brannock [24] to determine the chemical composition of major oxides from El Sela uranium ore material (Table 3). All of these samples are subjected to severe successive alteration processes.
Extraction conditions for thorium: Temperature and shaking time 5 min. Three representative samples were collected from El Sela thorium solution added to 10 ml of 0.04M TPPO dissolved in toluene in a separating funnel according to A/O ratio: 4/1. The content of separating funnel was shaking according to 5 min. at ambient room temperature (22±1ºC). After that, the mixture was separated and the loaded thorium on TPPO was stripped using 0.5M HNO₃ acid then thorium determined spectrophotometrically by Arsenazo III method.

Extraction conditions for uranium: Uranium concentration: 100 ppm, TPPO dissolved in toluene, Extractant conc. 0.02 M TPPO, A/O phase ratio 4:1, at ambient room temperature and shaking time 5 min.

While, the extraction of thorium (IV) was carried out by applying the previous factors controlling on thorium extraction. Take 40 ml of studied samples containing thorium solution added to 10 ml of 0.02M TPPO dissolved in toluene in a separating funnel according to A/O ratio: 4/1. The content of separating funnel was shaking according to 4 min. at ambient room temperature (22±1ºC). After that, the mixture was separated and the loaded thorium was stripped using 2M HNO₃ acid then thorium determined spectrophotometrically by Arsenazo III method.

The uranium concentrations in the studied samples ranged between 1100 and 6536 ppm. Meanwhile, the concentrations of uranium after stripping from TPPO by 0.5M HNO₃ acid indicated that, all studied samples were ranged between 1089 and 6525 ppm (Table 4).

While, thorium were ranged between 145 and 420 ppm. Meanwhile, the concentrations of thorium after stripping from TPPO by 2M HNO₃ acid indicated that, all samples were ranged between 132 and 402 ppm (Table 4).

4. Conclusion

Three representative samples were collected from El Sela area, south Eastern Desert of Egypt. The extraction of U (VI) and Th (IV) were tested using (TPPO) dissolved in different diluents. The parameters affecting the extraction and stripping of U and Th include type of diluents, TPPO concentrations, A/O phase ratio, shaking time, temperature, thermodynamic and stripping agents. The factors studied were followed by application of the suitable extraction system to extract U and Th from studied samples; El Sela area, south Eastern Desert, Egypt. It was found that the best extraction / stripping conditions for uranium were; 0.02M TPPO dissolved in toluene, A/O phase ratio 4:1, 5 min. of shaking time at ambient room temperature (22±1ºC) and 0.5M HNO₃ acid for stripping of uranium. Under these conditions, 99% of uranium is extracted from the studied samples.

On the other hand, for thorium extraction system the best conditions are; 0.04M TPPO dissolved in toluene, A/O phase ratio 4:1, 4 min. shaking time at ambient room temperature (22±1ºC) and 2M HNO₃ acid as stripping agent. For this system, the extractions (%) of thorium for all studied samples are ranged between 91.03 and 95.71 %.

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