Original Research Article

Chemical and Technical Investigations on Soil Characterization and Silicic Acid Preparation

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ABSTRACT

Physicochemical Characteristics of three soil samples collected from Qaser Bin Ghashir region, south of Tripoli, Libya have been investigated in terms of soil permeability, density (ρ), water content (WC), organic content (OC), dissolved salts content (DS), and inorganic ash content (IAC) as-well-as the reaction towards mineral acids and bases. Silicic acid Si $(OH)_4$ was successfully prepared from sandy soil sample in a considerable amount and the mechanistic equations were proposed for three possible preparation routes of the acid. Results show that sandy sample is the most permeable and reliable soil for the water penetration and silicic acid preparation respectively. Values of pH and the reaction between soil samples and mineral acids proof that the alkalinity nature of the samples. Chemical composition of the investigated samples were determined and were found to vary from the sample to another and from content to other according to the origin and nature of the soil.

Keywords: Soil, Physicochemical Characteristics, Permeability, Silicic Acid, Mineral Acids.

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Introduction

Soil is very important side of the triangle of any environmental ecosystem besides water and air. Soil was considered as the mother source of agriculture and food production chain for human and animal. Soil quality, soil type, the chemical composition and hence the soil production can affect greatly by different factors among of them the continuous uncontrolled cultivation, the irrigation behavior, weather/climate change and the environmental pollution. So, physicochemical characterizations of soil especially the local soil in Qaser Bin Ghashir region, south of Tripoli, Libya are increased in demand to ensure the validity and availability of soil for agriculture human activity in addition to the follow up of the soil environmental issues like chemical and pesticide contamination especially after the Libyan civilian war from 2011 to present. Soil has a variety or diversity in its composition; it contains mainly water, air, organic and inorganic materials (metals and metal oxides, non-metals and non-metal oxides, dissolved and non-dissolved salts) besides microorganisms and some living creatures. Some literature estimated that the percentage of inorganic minerals, air, water and organics in soil is about $\approx 45, 25, 25,$ and 5% respectively [1]. This percentage is not fixed for all types of soils; therefore may varies from type to another according to the weather or climate, the nature of the original rock source, and the human activities. Variation of chemical composition and characteristics of soil may lead to food production shortage, job loses that related to agriculture, and economic reduction, and hence returned to the total income of the country. The present research work aimed to evaluate the physicochemical characteristics of three different soil samples from Qaser Bin Ghashir region.

Materials and methods
Location and climate of the study area

Qaser Bin Ghashir region is located at an elevation of 73 meters above the Mediterranean Sea level and about 25 km far from the center of Tripoli, the capital of Libya. Its coordinates are 32º 40' 60" N and 13º 10' 60" E in DMS (Degrees, Minutes, Seconds) or 32.6833 and 13.1833 in decimal degrees [2]. Its UTM position is US21 and its Joint Operation Graphics Reference (JOGR) is NI33-13. It has a Mediterranean climate, characterized by a hot summer and a mild wintry temperature. The annual average temperature is 20°C. January and February are the coldest months (16-20°C on average). July and August are the hottest months (38-44°C on average). The relative humidity is about $\approx$ 65-84 % in winter and 25-35 % in summer [3]. Both geographical position and extremely warm weather in Qaser Bin Ghashir region support the agriculture activities as a result of the presence of various types of soil in this region where it contains huge wide area of cultivated lands such as farms, gardens, and wild plants in the large open area.

Soil samples

Three soil samples were collected from three different sites of the Qaser Bin Ghashir region, south of Tripoli, Libya in the winter 2019 during a heavy rain week. The average weight of each sample is approximately ≈ 1 kg. All collected soil samples were investigated initially by the nicked eye and found wet with the fall rain water as a result of higher moisture content. According to the source, soil samples were classified and categorized into the following three types:

- Sample number (1) which was designated by the cultivated soil that collected from a home cultivated garden in Qaser Bin Ghashir region.
- Sample number (2) which was designated by the combustible soil, where the sample was taken from the solid residual ash of a traditional homemade oven after combustion of the agriculture dry solid wastes (parts of plants like stems, roots, and leafs). The combustible soil sample was subjected into a traditional sieve to exclude the incombustible woody lumps of plants.
- Sample number (3) which was designated by the sandy soil that collected from unoccupied area (vacant) characterized by desert environment (has no plants) in around Qaser Bin Ghashir region.

Physicochemical analysis

Physical and chemical characterization of the three fresh soil samples were investigated in terms of density (ρ), water content (WC), organic content (OC), dissolved salts content (DS), and inorganic ash content (IAC). Density (g.cm^{-3}) of soil samples was determined using water displacement method as described in the literature [4]. Water content (WC) was determined by water evaporation or moisture loss method using drying oven at 105 ºC. Organic content (OC) and inorganic ash content (IAC) were determined by open air combustion method using a porcelain crucible [4]. Dissolved salts content (DS) was determined by dissolving a certain amount of soil sample in a desired amount of distilled water in a slightly acidic medium. Then the solution was filtrated and evaporated to dryness and the salty layer weighted. All the above mentioned methods depend upon the calculation of weight loss during evaporation, combustion, or dissolution. Each experiment was carried out in triplicates and the mean value was taken into consideration. All experiments were carried out under the laboratory temperature of ~ 16 ºC and the humidity is equal to ~ 80 %.

Permeability measurements

In a separate experiment, water permeability of the dry soil samples has been measured using a simple laboratory glass column with 120 mm height and 50 mm diameter as shown in Figure (1). The amount of water that passes through the soil under the gravity was collected in a graduated cylinder with time. Water permeability was measured and calculated in terms of milliliters per unit time (mL/min) or (mL/s).

Fig. 1: Determination of soil permeability using a glass column

Effect of acids and alkalis

The effect of mineral acids such as hydrochloric acid HCl, sulphoric acid H_2SO_4, and nitric acid HNO_3 also the effect of alkalis e.g. concentrated sodium hydroxide NaOH solution on the dried soil samples was studied to determine the type of dry soil sample. Above Experiment was undergoing using a 100 mL glass beaker. The reaction was relatively evaluated according to the color change, temperature change, effervescence, and evolved fumes.

Preparation of silicic acid

 A bench-scale experiment was carried out to prepare silicic acid from sandy soil sample number (3) after complete dryness. A pre-prepared 100 mL aqua regia solution (1V conc. HCl to 1V conc. HNO_3) was added to 50 gram of sandy soil sample in a 500 mL glass beaker under the fuming cupboard. Just the concentrated strong acidic solution was contacted to the sample; effervescence appeared with the evolution of condensed brown fumes due to oxidative degradation reaction of nitric acid with the organic materials of soil and therefore the ventilation system of the fuming cupboard was turned on to remove the accumulated hazardous gases (mainly N_xO_y gases). The reaction mixture was gently heated for about 5-7 minutes and the color of soil was changed to green, then the beaker was left for a week to complete the acid digestion process. The golden yellow color solution was filtered and neutralized by concentrated ammonia solution. A buff yellow color precipitate of silicic acid appears after neutralization and the excess ammonia was removed by heating the solution for 10 minutes. The precipitate was decanted and dried gently for two hours to produce the final product of silicic acid.

Mathematical forms

$$\text{Density} = (\rho) = \frac{m\ (g)}{\Delta v\ (cm^3)}$$

$$\%\ \text{Water Content} = \%\ (WC) = \frac{\Delta W}{W_i} \times 100$$

$$\%\ \text{Organic Content} = \%\ (OC) = \frac{\Delta W}{W_i} \times 100$$

$$\text{Inorganic Ash Content (g)} = (IAC) = W_t - [WC + OC]$$

$$\%\ \text{Inorganic Ash Content} = \%\ (IAC) = \frac{W_{IAC}}{W_t} \times 100$$

$$\%\ \text{Dissolved Salt} = \%\ DS = \frac{\Delta W}{W_i} \times 100$$

Results and Discussion

The obtained results of the present work contains three important parts, the first is the physicochemical characteristics of the soil samples under investigations. The soil properties were measured and evaluated according to the bulk density, water content, organic content, inorganic ash content, and dissolved salts in each individual sample. Permeability of the soil sample is the second special character that has been measured and evaluated practically. The third character of the soil samples upends upon the effect of the mineral acids and alkalis. All the above mentioned results give us a complete picture about the physical and chemical nature of the samples under investigation in addition to the ability to classify each sample to show the behavior of the soil with water and cultivated plants. In the following section, we will explore all of these physicochemical characteristics in details.

Physicochemical Characteristics and Permeability

The obtained results of density, (% WC), (%OC), (%IAC), (%DS), and permeability of the investigated soil samples were summarized in table (1). This table shows that all samples exhibit a relatively high density value ranged between the minimum value ≈ 2.2 g/cm^3 for cultivated soil and the maximum value ≈ 3.8 g/cm^3 for combustible soil sample. The differences in density values for the investigated samples reflect the diversity of chemical content in each sample. The density values are very important parameter for the classification of soil where the density and the behavior of soil towards the migration of water through different layers are strongly related and may reflect the irrigation requirements for individual soil type. The more density value of the soil, the low penetration ability of water will be and vice versa [5] and therefore someone expected

that the cultivated soil sample able to pass the water molecules faster than the other types of soil due to its relative low density value ≈ 2.2 g/cm^3. This low density value of cultivated soil may be explained by the fact that it contains the highest amount of the agriculture solid wastes like leafs, woody parts of the plants, and cellulosic materials of well-known low density values [6].

As a result of the combustion of organic materials in soil and the conversion of all soil materials into ash, the combustible soil sample has compacted very fine particulates and compacted layers, so the unit volume of this type contains the biggest number of particles and layers and hence the highest density value of ≈ 3.8 g/cm^3 and hence so difficulty for water molecules to penetrate the compacted particulates and layers. The vacancies between sand particles in sample number (3) either filled by the smallest sand particles or filled by irrigation water. In fact, there is no practical way to decide which rout actually occurs and favorite than the other, and therefore we need to know the real behavior of sandy soil by measuring the permeability character which can give us more details about how sands particles compacted with each other's in the sample? The high permeability value for the sandy soil (4.7 mL/s) proofs that the second rout is the predominant where the vacancies between sand's particles were filled with water stream. All samples under investigation show a relatively unusual high percent of water content due to the heavy rain on the region during the collection of samples and due to the accumulation of water along the rainy days.

Sample number (3) is the lowest water content and this may be explained by the high water permeability of the sample as a result of the course grain size of the sand granules. So, sandy soil passes the water molecules faster than the other samples instead of catching them. The grain size of the granules of sample number (1) and (2) are much smaller than sample number (3) and therefore the rate of diffusion of water stream is slow and catch much more water content, especially for cultivated soil sample due to the presence of clay. It was found that there are no organic content in combustible soil sample due to the conversion of organic matter into carbon dioxide gas (CO_2) during complete combustion and oxidation in open air. So, the ash residue contains only the naturally occurring inorganic metal and metalloid oxides like Na_2O, K_2O, MgO, CaO, Fe_xO_y, ZnO, Al_2O_3 and SiO_2 [7]. The high reasonable organic content of ≈ 14.8 % was found in cultivated soil, so it can be described as organic rich soil while sample number (2) described as organic poor soil. The source of organics in sample (1) and (3) is the animal or/and agriculture wastes which includes nitrogen (N) as a source of protein, peptides, and amino acids besides carbon (C) and sulpher (S). Cultivated soil is organic rich sample and this may be attributed to the fact that organic materials are much more durable than the inorganics by continuous cultivation [8]. The highest inorganic content of ≈ 84.7 % was recorded for sample number (2), whereas the lowest inorganic content was observed for the cultivated soil sample. The natural source of inorganics in cultivated and sandy soil samples is rocks (igneous, metamorphic, and sedimentary) and minerals (mainly olivine, muscovite, feldspar, and quartz) [9]. Also it was observed that the dissolved salt (DS)

content is in a minimal amount of less than ~ 1-2 % and it can be explained by the soil washing or soil leaching process during the heavy rain days.

The dissolved salts like halite or rock salt (NaCl), tarona ($Na_2CO_3.2NaHCO_3.3H_2O$), natron ($Na_2CO_3.H_2O$), and carnalite ($KCl.MgCl.6H_2O$) are natural soluble minerals and can dissolved easily in the heavy rain water and consequently go away from the mother soil material as a result of soil erosion. The natural insoluble minerals includes mainly the silicates minerals such as feldspar [$K_2O.Al_2O_3.6SiO_2$], mica [$KH_2Al (SiO_4)_3$], kaolin [$Al_2O_3.2SiO_2.2H_2O$], gibbsite [$Al_2O_3.3H_2O$], and bauxite [$Al_2O_3.2H_2O$] [10]. Soil permeability is a measure indicating the ability of the soil allows fluids (mainly natural water) to pass through it. Soil permeability represented by the permeability coefficient *(K)* in the Darcy's equation: $V = K \times i$, where *V* is the apparent fluid velocity through the medium in m/s, and i is the hydraulic gradient, and *K* is the coefficient of permeability (hydraulic conductivity) often expressed in m/s. K depends on the relative permeability of the medium for water and the dynamic viscosity of the fluid as follows:

$$K = \frac{\gamma_w \times \beta}{\eta}$$

Where γ_w is the unit weight of water, η is the dynamic viscosity of water, and β is an absolute coefficient depending on the characteristics of the medium. Practically the coefficient of permeability *(K)* can be calculated using the following formula [11]:

$$K = \frac{q \cdot L}{A \cdot h}$$

Where *q* the amount of discharged water in cm^3/s or mL/s, *L* is the length of specimen in cm, *A* is the cross sectional area of the soil specimen in cm^2 and *h* is the constant head causing water flow. According to the date shown in Table (1) the coefficient of permeability (hydraulic conductivity) of the investigated three soil sample can ordered in the following sequence:

<div align="center">Sandy soil > Cultivated soil > Combustible soil</div>

This can be attributed to the differences between samples in its physicochemical characteristics of each soil such as the grain size, density, and the chemical constituents.

Table 1: Physicochemical characteristics of the investigated soil samples

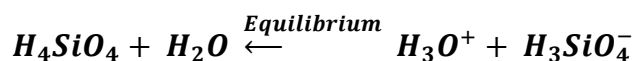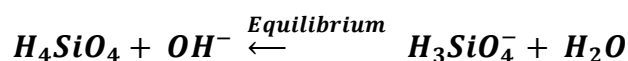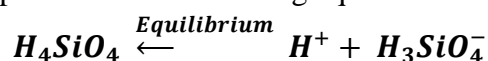| Property	Sample (1) (Cultivated Soil)	Sample (2) (Combustible Soil)	Sample (3) (Sandy Soil)
Color	Mud color	Gray color	**Pale yellow**
Density	2.2 g/cm^3	3.8 g/cm^3	**2.5 g/cm^3**
% Water	17.6 %	15.3 %	**10.5 %**
% Organic	14.8 %	0 %	**10.8 %**
% Inorganic	67.6 %	84.7 %	**78.7 %**
% Soluble Salts	N.A	~ 2 %	**<% 1**
Permeability	3.3 mL/s	2.3 mL/s	**4.7 mL/s**

| pH-value | 9.11 | 9.54 | **8.93** |
| Quantity of Silicic acid salt | N.A | N.A | **15.3 g (30.6 %)** |

N.A: Not Available

Preparation of silicic acid

Silicic acid or ortho-silicic acid, or tetrahydroxysilane, or silicon tetrahydroxide has the chemical formula [H_4SiO_4 or $Si(OH)_4$] as shown in figure (2) is pure inorganic weak acid. Silicic acid was prepared practically as mentioned in the experimental section using acid digestion method by aqua regia solution. The prepared silicic acid has a gelatinous or colloidal character as shown in Figure (3), so it flows like oil. As shown in Figure (3), silicic acid is a buffy, light dusty, and has a pall yellow color. Silicic acid is a monomer in a fluid phase; however in the solid state these probably condense to form polymeric silicic acids of cross-linked 3D complex structure as shown in Figure (4) [12]. Although silicic acid is a polyprotic acid, it is a very weak acid and acts as a mono-basic acid, so it donates one proton as expressed in the following equilibrium equations:
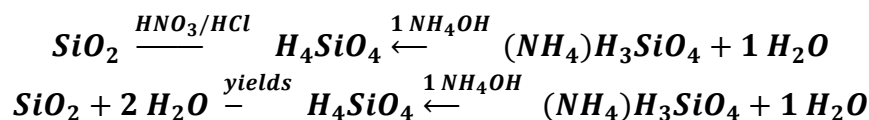
$$H_4SiO_4 \xleftarrow{Equilibrium} H^+ + H_3SiO_4^-$$

$$H_4SiO_4 + OH^- \xleftarrow{Equilibrium} H_3SiO_4^- + H_2O$$

$$H_4SiO_4 + H_2O \xleftarrow{Equilibrium} H_3O^+ + H_3SiO_4^-$$

$$K_a = \frac{[H_3O^+][H_3SiO_4^-]}{[H_4SiO_4]}$$

According to the literature [13], the value of ionization/dissociation or equilibrium constant *(K_a)* of silicic acid is equal to 1.318×10^{-9} and the *(Pk_a)* value is equal to (8.88 ± 0.15) at 130 ℃ which indicates that it is a weak inorganic acid.
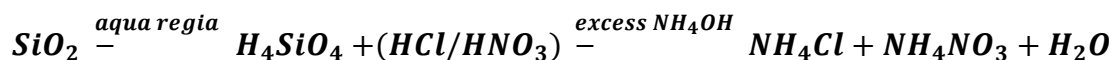
The mechanistic equations for the preparation of silicic acid can be represented as follows:

(1) The first possible route: the mono-basic ammonium silicilate formation. The ammonium salt of silicic acid is derived from a mono weak basic radical (NH_4^+) and a mono weak acidic radical ($H_3SiO_4^-$), so it is unstable salt that can undergo an equilibrium with silicic acid itself.

$$SiO_2 \xrightarrow{HNO_3/HCl} H_4SiO_4 \xleftarrow{1\,NH_4OH} (NH_4)H_3SiO_4 + 1\,H_2O$$
$$SiO_2 + 2\,H_2O \xrightarrow{yields} H_4SiO_4 \xleftarrow{1\,NH_4OH} (NH_4)H_3SiO_4 + 1\,H_2O$$

(2) The second possible route: silicic acid formation followed by neutralization of excess aqua regia.

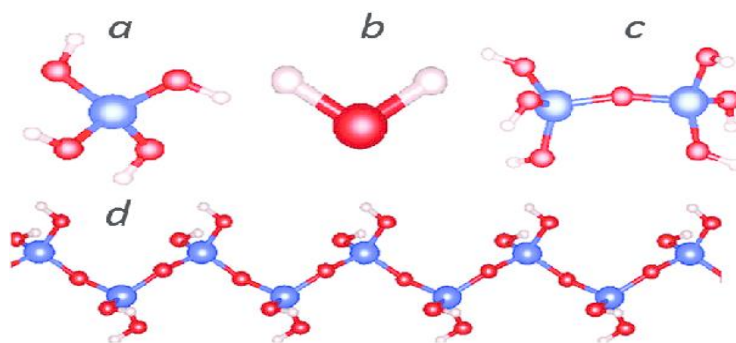$$SiO_2 \xrightarrow{aqua\ regia} H_4SiO_4 + (HCl/HNO_3) \xrightarrow{excess\ NH_4OH} NH_4Cl + NH_4NO_3 + H_2O$$

(3) The third possible route is the mixed or hybridized mechanism: in this proposed mechanism, both the first and the second above routes will occur simultaneously, so silicic acid H_4SiO_4, its ammonium salt $(NH_4)H_3SiO_4$ and the two neutralization products (NH_4Cl & NH_4NO_3) will present in the reaction mixture. However ammonium chloride (NH_4Cl) and ammonium nitrate (NH_4NO_3) are soluble simple salts, and hence it surly found in the aqueous phase whereas silicic acid H_4SiO_4 and its ammonium salt $(NH_4)H_3SiO_4$ were present in the gelatinous condensed lower layer. Almost chemical reactions were preceded via more than one single pathway with different extent to produce the main product besides byproduct, so the mixed or hybridized mechanism is the most probable pathway for the preparation of silicic acid as the main product.

Fig. 2: The Chemical Structure of Silicic acid

Fig. 3: Practically Prepared Silicic Acid

Fig. 4: Monomer, dimer, and cross-linked silicic acid [12]

a: Monomer   b: Water   c: Dimer   d: Cross-linked

Effect of mineral acids and alkali

The effect of concentrated solutions (> 5M) of sulphoric acid, nitric acid, hydrochloric acid, and sodium hydroxide on the three investigated soil samples has been studied and evaluated in terms of the presence of effervescence, color change, evolution of water vapor, evolution of chemical fumes, and the raise of temperature. The obtained results are presented in Table (2), from which we can observed that all soil samples were affected greatly by the mineral acids whereas all soil samples never affected by sodium hydroxide solution at all. This observation indicates that all soil samples are alkaline in nature due to the presence of naturally occurring calcium and magnesium rich minerals such as: calcite/limestone, chalk, marble/stone $CaCO_3$, gypsum $CaSO_4.2H_2O$, natural phosphorite $Ca_3(PO_4)_2$, manmade synthetic rock $Ca_3(PO_4)_2$, magnesite $MgCO_3$, dolomite $MgCO_3$. $CaCO_3$, asbestos $CaMg_3(SiO_3)_4$, Epsom salt $MgSO_4.7H_2O$, and soapstone (talc) $Mg_3H_2(SiO_3)_4$ [14 & 15]. Calcium and magnesium in naturally occurring minerals were found as carbonate, sulphate, phosphate and silicates. The alkaline nature of soil samples was confirmed by the pH values in Table (1) where the pH values are ranged between $9.54 > pH > 8.93$. The effect of mineral acids on soil samples was varied from strong effect (+++) for Cultivated sample, mild or medium effect (++) for sandy soil, and finally weak effect (+) for combustible soil sample and this behavior may be understood in the light of the fact that each type of soil has its own characteristics and its own chemical composition. From the chemical point of view, effervescence occurs as a result of evolution of both water vapor and chemical fumes that can produced as a result of elevated temperature and oxidation of organics by mineral acids respectively. Also the color change and the raise of temperature were occurred as a result of oxidation of organic materials in samples. Oxidation of organics by mineral acids produces CO_2 gas, whereas the reaction of mineral acids with alkaline minerals may produces SO_2, SO_3, H_2S, N_xO_y gases. Simultaneous evolution of water vapor, CO_2 gas, offensive sulpher gases, and brown colored nitrogen gases leads to strong Effervescence. The maximum temperature ($T_{max.}$) of the reaction between individual soil sample and each acid solution was recorded in (ºC) and the date in Table (2) shows that the maximum temperature was appeared with

the cultivated soil followed by sandy soil and finally with Combustible Soil. This result was in agreement with the data in Table (1) where cultivated soil has the highest percentage of water and organic contents leads to the vigorous evaporation and oxidation reaction and hence the raise of temperature will be. The data shows that the relative oxidative power of the sulphoric and nitric acids are seem to be approximately the same, but hydrochloric acid has the low oxidative power on soil samples. This may be due to the fact that oxidative power of the oxy-acids is higher than the non oxy-acids [16 & 17].

Table 2: Effect of acids and alkali on soil samples

Property	Sample (1) (Cultivated Soil)				Sample (2) (Combustible Soil)				Sample (3) (Sandy Soil)			
	H_2SO_4	HNO_3	HCl	NaOH	H_2SO_4	HNO_3	HCl	NaOH	H_2SO_4	HNO_3	HCl	**NaOH**
Effervescence	+++	+++	+++	-ve	+	+	+	-ve	++	++	++	**-ve**
Color change	+++	+++	+++	-ve	+	+	+	-ve	++	++	++	**-ve**
Water vapor	+++	+++	+++	-ve	+	+	+	-ve	++	++	++	**-ve**
Fumes	+++	+++	+++	-ve	+	+	+	-ve	++	++	++	**-ve**
Raise of temperature	+++	+++	+++	-ve	+	+	+	-ve	++	++	++	**-ve**
$T_{max.}$ (ºC)	97	96	68	-ve	30	30	26	-ve	71	69	42	**-ve**

+++ strong effect   ++ Mild effect   + Weak effect   -ve Negative effect   Room temperature ~ 16 ºC

Conclusions

Three different soil samples from Qaser Bin Ghashir region, 25 Km south of Tripoli, Libya have been characterized in terms of permeability, density, water content, organic and inorganic content. The obtained results proof that each sample has its own characteristics and silicic acid can be prepared from the sandy soil only. Investigated soil samples were affected greatly by mineral acids and the reaction is vigorous, so all samples exhibit alkaline property especially, cultivated soil.

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