# Evolution of the Carpometric Characteristics and the Chemical Composition of Oils During the Period of Maturity of the Olive in the Chaouia Area -Morocco

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*Abstract* - Several factors affect the quality and quantity of the olive oil, such as cultivation techniques, fluid intake, period of harvest, storage conditions and extraction techniques. The optimization of the quality of olive oil requires a relevant determination of the olive harvest period. With the aim of determining this period, a follow-up of the evolution of different parameters namely: the carpometric characteristics, the maturity index, the water content, the content of oils and chemical composition of olive oil in two different sites in the Chaouia area (in the center of Morocco) were carried out since the month of June until the month of January.

The results showed that the weight, dimensions, free acidity and the oil content increase with the maturity of the olives and reach their maximum values in December. Inversely, the contents of chlorophyll, water and polyphenols that were maximal at the beginning of fructification tend to decrease with the maturity of the fruit. The maturity index which was null during the green stage olives, increases with maturity. The analysis of the acidic composition shows that the palmitic acid and oleic acid levels decrease during maturation; however, that linoleic acid increases. The combination of these parameters was used to determine an optimum harvesting time for olives in this area of Morocco to obtain an oil of good quality. For this campaign (2013-2014), the optimal date of harvest of olives is fixed to mid-December.

Keywords: Optimal date of harvest of olives, carpometric characteristics, maturity index, chemical composition of olive oil.

# I INTRODUCTION

Olive oil is one of the principal component of the diet of the communities in the Mediterranean. It contains a large number of compounds; the main ones are the triacylglycerols, fatty acids [1], and a large number of minor components present in small quantities such as phenols, tocopherols, sterols, hydrocarbons, pigments and aromatic compounds [2,3]. This chemical composition and consequently the quality of olive oil depends essentially on intrinsic factors such as varietal factor [4,5], extrinsic

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factors such as the maturity of the olives, the cultivation techniques and the mode of extraction and conservation of the oil [6,7].

The optimization of the quality of olive oil requires a judicious determination of the period of determining this period, a monitoring of the evolution of different parameters namely: the carpometric characteristics, the chemical composition of the oil from two sites in the Chaouia area was carried out over a period harvest of the olive [8,9]. With the aim of of seven months.

# II MATERIALS AND METHODS

# 1 - Olive fruit sampling

This study was carried out in the Chaouia area (center of Morocco) during the crop seasons 2013 / 2014. It interested two sites: Oulad Said (site 1) and Sidi el Aïdi (site 2). The plant material studied belongs to the variety Moroccan Picholine population. An olive sample of 1 kg was collected from trees of two sites, regularly, over a period of seven months (from June 20 th until January 20 th).

# 2 - Determination of dimensions and weight of olives

Of 100 randomly chosen fruit, one determines the length and width of fruit by a caliper. The core weight and that of the whole fruit is determined by weighing with a precision balance. [10].

# 3 - Determination of the maturity index of olives

The maturity index (MI) was determined by the visual appreciation of the color of the fruit in a color scale ranging from the dark green to black and a flesh entirely dark [11,12]. It is calculated according to the following formula:

MI = (a x 0) + (b x 1) + (c x 2) + (d x 3) + (e x 4) + (f x 5)+ (g x 6) + (h x 7) / 100 0 < MI < 7

Where a, b ... h are the numbers of the olives in each of the following numerical classes:

0 =skin is a deep or dark green colour.

1 =skin is a yellow or yello wish-green colour.

2 = skin is a yellowish colour with reddish spots.

3 =skin is a reddish or light violet colour.

4 =skin is black and the flesh is completely green.

5 = skin is black and the flesh is a violet colour halfway through.

6 = skin is black and the flesh is a violet colour almost through to the kernel.

7 =skin is black and the flesh is completely dark.

#### 4 - Determination of water content:

A sample of whole fruit, of known weight, was ground in a blender then dried in an oven heated to  $105 \degree$  C until obtaining constant weight [13]. The dry weight of the sample was determined and the water content was then calculated.

# 5 - Chemical characterization of olive oil

# 1 -5 Extraction of olive oil and determination of its content:

In order to neutralize lipases, enzymes able to degrade triglycerides, each sample of fruit was put into boiling water during 5 minutes. The olives were ground dry and the oil is extracted in two consecutive times by the hexane. After the evaporation of hexane to the rotary evaporator, the oil content is then determined. It is expressed as a percentage (g oil / 100g of olives).

# 2 -5 Determination of free acidity

The free acidity, expressed as a percentage of oleic acid, was determined by 1 g of olive oil dissolved in 50 ml of ethanol. The mixture was titrated by a solution of potassium hydroxide 0.1 N in the presence of phenolphthalein [14].

#### 3-5 Determination of chlorophyll contents

5 ml of olive oil dissolved in 5 ml of carbon tetrachloride. After homogenization, the absorbances were measured at 670, 630 and 710 nm [15]. The chlorophyll content is calculated using the following formula:

Chl (ppm) = 
$$\frac{A_{670} - \frac{(A_{630} + A_{710})}{2}}{0,1086}$$

4 - 5 Determination of content in phenolic compounds

The total phenolic compounds, contained in the studied oils, have been extracted in the mixture methanol-water 80:20 (v / v). The upper phases obtained after centrifugation were collected and the dosage is carried out

by the method of Folin-Ciocalteu reactive at 750 nm in the presence of a standard range of caffeic acid [16].

#### 5 - 5 Analysis of the acidic composition

The methyl-esters of the fatty acids are obtained by the action of methanol in alkaline medium of glycerides and free fatty acids, according to the standard method recommended by the International Olive Oil Council [17]. To 0,1 g olive oil one adds 2 ml of heptane and 0,2 ml of of 2N methanolic potash. After agitation during 30 seconds, the heptanic higher phase is recovered.

The methyl esters of fatty acids obtained were analyzed by gas chromatography HP (model 6890) equipped with a flame ionization detector (T =  $260 \degree$  C). The column used is a capillary column Carbowax type size 30 m x 0,32 mm x 0,25 µm. The carrier gas was nitrogen at a flow rate of 2,5 ml / min. The oven temperature program was 140 ° C to 200 ° C, from 210 to 245 ° C with a gradient of 10 ° C / min every 10 min. The identification of the peaks was carried out in the presence of standard compounds and various percentages of fatty-acids were calculated by means of an automatic integrator.

All the analyzes were carried out in triple and the results are expressed by averages and standard deviations.

# III RESULTS AND DISCUSSION:

1 - Study of carpometic parameters during the maturity of olives

#### 1-1 Evolution of the weight of olives:

The evolution of the weight of olives from the two studied sites is represented in Figures 1 and 2.

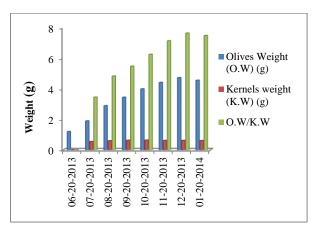


Fig 1: Evolution of the weight of olives for the site of Oulad Said.

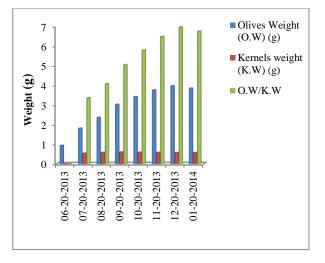


Fig 2: Evolution of the weight of olives for the site of Sidi el Aïdi

We note that during the month of June, the kernel weight is zero for both sites. Towards the end of July, the kernel hardens and its weight is  $0.55 \pm 0.08$  g for site 1 and  $0.54 \pm$ 0,09 g for site 2. On August 20th, the weight of the kernel reached  $0.6 \pm 0.07$  g for site 1 and  $0.58 \pm 0.08$  g for site 2. After this date, the weight remains practically unchanged during the maturity of the fruit. Indeed, the kernel is formed by the endocarp which sclerified to protect the embryo (seed). This sclerification blocks the metabolic phenomena and allows the seed to remain in state of latent life for long periods [18], which explains the almost constant weight of the kernel. Compared to the literature, our results agree with those obtained by Desouky et al. in Egypt who found a kernel weight equal to 0,58 g for the variety Arbequina. [19] In Morocco, for the same variety Moroccan Picholine, Ajana et al. found a value of 0,55 g in the Marrakech region [20].

Concerning the weight of the fruit, there is an increase between June and December when the maximum values are reached. Indeed, for the sites of Oulad Said and Sidi el Aïdi, fruit weight increased from  $1,2 \pm 0,3$  to  $4,73 \text{ g} \pm 0,2 \text{ g}$  and  $0,94 \pm 0,2 \text{ g} 3,96 \pm 0,2 \text{ g}$  respectively. This increase in weight represents the phase of growth of the fruit which is accompanied by a multiplication and intense growth of the mesocarp cells. From December 20 th to January, the weight begins to decrease. The latter is caused by the loss of water from the fruit through transpiration [21].

Furthermore, the report of the weight of the fruit on the weight of the kernel follows the same evolution of the weight of the fruit since the weight of the kernel remains unchanged for the period of maturity of the olives. It passes during the period from July to December, from 3,45 to 7,63 for site 1 and 3,35 to 6,95 for site 2. In January this ratio decreases. It passes from 7,63 to 7,48 for site 1 and 6,95 to 6,74 for site 2.

However, the values of weight observed in this study are lower than those reported by Keceli, which obtained the values 6.4 g for variety Topagi Adana and 5.9 g for variety Gemlik in Turkey [22] and higher compared to those reported by Desouky et al. which found values 2,30 g, 1,24 g and 1,21 g respectively, for the varieties Arbequina, Koroneiki and Bouteillan in Egypt [19].

Essiari and al. reported similar values for the same variety ( Moroccan Picholine) in Sais region (4,43 g) **[23]**. In addition, Rahmani noted that the weight of the olives of the Moroccan Picholine variety oscillates between 3 and 5 g **[24]**.

# 1-2 Evolution of fruit dimensions

Figures 3 and 4 represent the dimensions of the olives (length (L) and width (l)) taken at different stages of maturity in both sites.

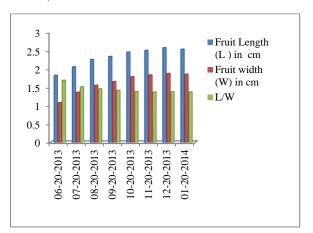


Fig 3: Evolution of olives dimensions (expressed in cm) taken for the site of Oulad Said.

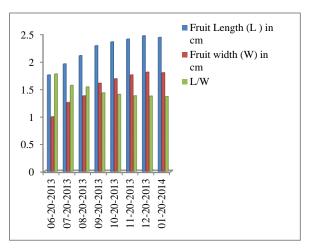


Fig 4: Evolution of olives dimensions (expressed in cm) taken for the site of Sidi el Aïdi.

According to Figures 3 and 4, one notices an increase in fruit length from the month of June to December when the maximum values are reached: the values have increased from  $1,82 \pm 0,3$  cm months June to  $2,57 \pm 0,1$  cm and 1,75 cm  $\pm 0,4$  to  $2,46 \pm 0,1$  cm for site 2. In the month of January the fruit reduces the length of a few millimeters to achieve  $2,53 \pm 0,2$  cm for site 1 and  $2,43 \pm 0,2$  cm 2 for site 2.

Similarly, we note that the width of the fruit increases with the maturity of the olives. It passes from  $1,08 \pm 0,2$  cm in

the month of June to reach a maximum  $1,87 \pm 0,1$  cm in December for site 1 and  $0,99 \pm 0,1$  cm to  $1,8 \pm 0,1$  cm for site 2. In the month of January, the fruit width decreases. It reached a value of  $1,85 \pm 0.1$  cm for site 1 and  $1,79 \pm 0,1$ cm 2 for site 2. The increase in the length and width of the fruit is a consequence of the increase in its weight.

As to the relationship between the length to the width, it decreases during the period from the month of June until the month of November for the two sites. It passes from 1,69 to 1,37 for site 1 and 1,77 to 1,36 for site 2. This decrease is due to the growth of the mesocarp causing a greater increase in the width than in the length. From November 20 th to January 20th, this report remains unchanged and equal to 1,37.

Ait Yassine reported similar values for olives belonging to the same variety (Moroccan Picholine) of the Tadla area. She found values ranging from 1,6 to 1,8 cm in width and 2,2 to 2,45 cm in length **[25]**. For olives produced in the area of Marrakech, Ajana et al. found values oscillating between 0,96 to 1,69 cm in width and 1,74 to 2,34 cm in length **[20]**.

#### 1-3 Determination of the maturity index of olives

The results of the maturity indices obtained for different samples of both sites are represented by the following table:

Table 1: Evolution of the index of maturity of both studied sites.

Dates of sampling	Site 1	Site 2
20-June	0	0
20-July	0	0
20-August	0	0
20-September	0,32	0,41
20-October	1,37	1,42
20-November	2,77	2,92
20-December	3,78	3,9
20-January	5,32	5,61

According to table 1, one notes that for the first three months (June, July and August), the maturity index is null. During these months, the color of olives is intense green. Toward September, the olive maturity index starts to increase. Indeed chlorophyll is transformed, degraded or destroyed to other pigments like pheophytins and anthocyanins [26]. These are the pigments that are responsible for the purple and black color of olives. The black color of olives is also due to the oxidation of phenols in particular oleuropein [27].

These results are in complete agreement with those found by Ait Yassine in the area of Tadla [25].

#### 1-3 Water content of the olives

The evolution of the water content of olives compared to the fresh matter for both sites is represented by Figure 5:

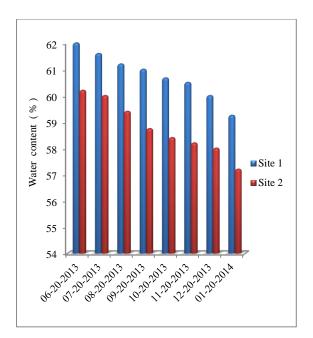


Fig 5: Evolution of the water content compared to the fresh matter (expressed in the percentage) of olives for the two sites studied.

The presence of water is essential for the life of the fruit cells since all biochemical reactions occur in aqueous environment. There can be no cell division, thus, no growth and development of the fruit without water.

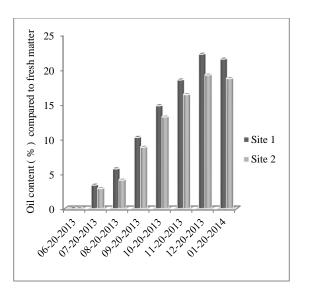
The results of the evolution of the olive water content compared to the fresh matter for the two sites studied show that this content is important and represents more than 50% of the weight of the olive. It varies between  $62 \pm 0.5$  %  $59,25 \pm 0.7$  % for site 1 and  $60,2 \pm 0.7$  to  $57,2 \pm 0.9$  %. This water content is influenced by hydrous contribution (pluviometry, irrigation) and by temperature.

Compared to the literature, our results are in agreement with the values found by other authors in Australia [28] and Jordan [9].

# IV - CHEMICAL CHARACTERIZATION OF THE OIL DURING THE MATURITY OF THE OLIVES

#### 1 - Oil content of olives

Figure 6, represents the evolution of the oil content of olives compared to the fresh matter of both sites:



We note that the values of the oil content are null during the month of June. The synthesis of oil has not yet started.

Fig 6: Evolution of the oil content of olives of the two sites.

It is from July that the lipogenesis is begins and we note the presence of a small quantity of oil  $(3,4 \pm 0,3\%)$  for site 1 and  $2,9 \pm 0,4\%$  for Site 2). The synthesis of oil continued and reached maximum values during the month of December (22,2 0,3\%) for site 1 and 19,2 0,3\%) for site 2). Beyond that date, there was a slight decrease in oil content.

The values which we obtained are in agreement with those reported by Ajana et al. in the area of Marrakech [20] and Ait Yassine in the area of Tadla [25]. Loussert et al. [29] and Walali et al [12] noted that Moroccan Picholine contains a quantity of oil ranging from 19 to 25 %. This oil content is influenced by several factors such as the variety, the climate, and the cultural practices [30].

# 2 - Evolution of free acidity during the maturity of the olives

The analysis of the free acidity of oils of the two studied sites yielded the following results. They are expressed in the percentage of oleic acid (Table 2):

Table 2: Percentage of oleic acid in a	olive oils: Site of Ouled Said.
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Dates of sampling	20-July	20-August	20-September	20-Octobrer	20-November	20 Décember	20-January
free acidity	0,15 ± 0,01	0,2 ± 0,015	0,26 ± 0,01	0,3 ± 0,01	0,35 ± 0,01	0,4 ± 0,01	0,45 ± 0,01

Table 3: Percentage of oleic acid in olive oils (site of Sidi el Aïdi,)

Dates of sampling	20-July	20-August	20-September	20-Octobrer	20-November	20 Décember	20-January
free acidity	0,19 ± 0,01	$\textbf{0,24} \pm \textbf{0,01}$	0,3± 0,01	$\textbf{0,33} \pm \textbf{0,01}$	$\textbf{0,39} \pm \textbf{0,01}$	0,42 ± 0,01	$\textbf{0,49} \pm \textbf{0,01}$

We notice that the free acidity increases with the maturity of the olives. It varies between 0,15 and 0,45 % for the site of Ouled Said and between 0,19 and 0,49 % for the site of Sidi el Aïdi.

These results are close to the values found by Al-Maaitah et al. for the Jordanian olive oils that oscillate between 0,19 and 0,66 % [9] but lower than those reported by Desouky et al. who have obtained values between 0,6 and 1 % for olive oils from Egypt. [19]. For the olive oils produced in Turkey, Keceli found values ranging from 0,63 to 1,15%. [22].

# 3 - Evolution of the chlorophyll content

The contents obtained for chlorophylls, samples studied for the two sites, expressed in ppm, are represented by the following figure:

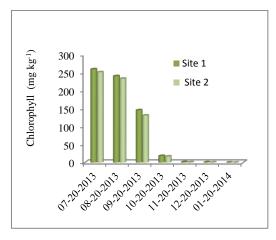


Fig 7: chlorophyll content of samples studied for the two sites.

According to the results obtained, it is noted that the chlorophyll concentration is high during the first samples of olives. It was of the order of  $259,25 \pm 0,02$  ppm for site 1 and  $251 \pm 0,03$  ppm for site 2. This concentration

gradually decreases with the maturity of the olives to achieve very low values of the month of January (0,12  $\pm$  0,02 ppm for site 1 and 0,16  $\pm$  0,02 ppm for site 2). This decrease is due to the degradation of chlorophyll of pheophytins which confers the oil its yellow color [26].

The results found are in agreement with those found by Aslan et al in Turkey [31].

# 4 - Evolution of the content of phenolic compounds

The evolution of the contents of the phenolic compounds, samples studied for the two sites, expressed in ppm, are represented by the following figure:

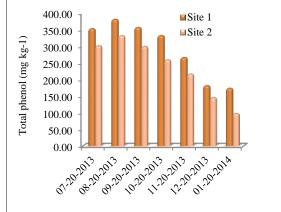


Fig 8: Content of phenolic compounds studied samples for both sites.

The evolution of the total polyphenol content of the oil shows important values during the months of July and August (from 352,66 to  $380,88 \pm 0,03$  ppm for site 1 and 301,60 to  $331,80 \pm 0,02$  ppm for site 2). This content tends to decrease with the maturation of the olives to reach lower values during the month of January:  $173,98 \pm 0,02$  and  $97,40 \pm 0,02$  ppm respectively for the site1 and site 2. Indeed, the polyphenols are natural antioxidants whose function is the protection of olive oil against oxidation. The content of phenolic compounds is variable. It is influenced by several factors: the variety of olive and its maturity, method of extraction and storage of the finished product **[32].** 

The values found in this study are higher than those found by Ait Yassine for the same variety (Moroccan Picholine) in the area of Tadla [25]. She found values ranging from 33,86 to 0,06 ppm.

# 5 - Evolution of the acidic composition of olive oil

The results of the gas chromatography are shown in the tables below:

	20-July	20-Aug	20-Sep	20-Oct	20-Nov	20-Dec	20-Jan
Palmitic acid C16:0	14,83	14,54	13,55	12,54	9,61	8,64	8,97
Palmitoleic acid C16:1	0,89	0,87	1,18	0,9	0,73	0,65	0,75
stearic acid C18:0	1,71	1,73	2,52	1,63	2,33	2,48	2,5
oleic acid C18:1	73,2	73,52	74,01	71,55	73,65	74,42	73,61
linoleic acid C18:2	6,37	6,22	6,44	11,49	11,91	12,19	12,73
linolenic acid C18:3	2,15	2,27	1,62	1,16	0,92	0,84	0,77
Arachidic acid C20:0	0,34	0,34	0,45	0,31	0,26	0,28	0,39
Gadoleic acid C20:1	0,36	0,33	0,23	0,43	0,37	0,34	0,45
SFA	16,88	16,61	16,52	14,48	12,2	11,4	11,86
UFA	82,97	83,21	83,48	85,53	87,58	88,44	88,31
UFA/SFA	4,92	5,01	5,05	5,91	7,18	7,76	7,45

Table 5: Percentage of fatty acid of the oils studied site 1.

	20-July	20-Aug	20-Sept	20-Oct	20-Nov	20-Dec	20-Jan
Palmitic acid C16:0	14,64	14,83	13,65	12,58	10,1	9,92	9,18
Palmitoleic acid C16:1	0,85	0,88	1,04	0,67	0,73	0,86	0,66
stearic acid C18:0	1,7	1,72	1,5	1,62	2,13	1,98	2,12
oleic acid C18:1	74,99	73,36	74,85	72,83	71,83	69,56	72,84
linoleic acid C18:2	5,26	6,06	6,46	10,27	13,54	15,98	12,94
linolenic acid C18:3	1,77	2,25	1,41	0,98	0,77	0,93	0,92
Arachidic acidC20:0	0,29	0,4	0,52	0,33	0,35	0,36	0,43
Gadoleic acid C20:1	0,34	0,36	0,57	0,41	0,43	0,4	0,59
SFA	16,63	16,95	15,67	14,53	12,58	12,26	11,73
UFA	83,21	82,91	84,33	85,16	87,3	87,73	87,95
UFA/SFA	5,00	4,89	5,38	5,86	6,94	7,16	7,50

Table 6: Percentage of fatty acid of the oils studied site 2.

The results obtained for the various oils studied show that the majority fatty acid is monounsaturated oleic acid (C18: 1), followed by linoleic acid and palmitic acid. The minor fatty acids, whose percentages do not exceed 3%, are represented by palmitoleic acid, stearic acid, linolenic acid, arachidic acid and gadoleic acid.

The evolution of the content of these fatty acids during the maturity of olives is not the same. Indeed, the oleic acid content, for site 1, remained practically stable (the content varies between 73,65 % and 74,01 %), while for site 2, the content decreased. It passed from 74,99 % in June to 69,56 % in December. In January the content increased to reach the value 72,84 %.

The content of palmitic acid decreased for both sites while that of linoleic acid increased. These two fatty acids are developing in an opposite way. By contrast, palmitoleic acid and gadoleic acid have the same evolution: the values are low in the month of June and they increase to reach maximum values in October, then they decrease towards the end of the maturity of olives.

The presence of the ponlyinsaturé fatty acid: linoleic acid (C18: 2) with an important percentage compared to other unsaturated fatty acids can be explained by the presence of an enzyme, oleate denaturase wich transforms the oleic

acid (C18: 1) ) in linoleic acid (C18: 2) during the fruit ripening [**33**].

The fatty acid composition obtained reveals a predominance of monounsaturated fatty acids. The percentage of unsaturated fatty acids (UFA) increases with the maturity of olives. It varies between 82,97 % and 88,31 % for site 1 and 83,21 to 87,95 % for site 2. However, the percentage of saturated fatty acids decreases, it passes from 16,88 to 11, 86 % for site 1 and 16,36 % to 11,73% for site 2.

The relationship between unsaturated fatty acids on saturated fatty acids (UFA / SFA) shows consequently a clear increase with the maturity period. This increase is

very high, which gives the olive oil greater stability to auto-oxidation and a high nutritional value [34,35].

# CONCLUSION :

The follow-up of the evolution of physicochemical characteristics and acidic composition of olive oils the variety Moroccan Picholine in the area of Chaouia since the month of June until January enabled us to conclude that the weight of fruits, their dimensions, the maturity index, free acidity and olive oil content increase with maturity.

The study of all these parameters allowed us to conclude that for obtaining a good quality of olive oil, the olive harvest must be at about the middle of December for several reasons: the weight, dimension and oil content of olives are maximum, the contents of chlorophyll and polyphenols are appreciable and the monounsaturated fatty acids are predominant.

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