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Valorization of apricot, melon, and watermelon by-products by extracting vegetable oils from their seeds and formulating margarine☆

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Abstract – Vegetable oils extracted from seeds and oleaginous fruits are a substantial source of bioactive compounds. In this study, oils of some fruit by-products were investigated and their composition and properties were compared. Apricot (ASO), melon (MSO), and watermelon (WSO) seed oils were extracted by cold pressing. The physico-chemical parameters and the contents of pigments were assessed using standard methods. The values of the physico-chemical parameters revealed the purity of the oils and it was recorded that the WSO has the best contents of chlorophylls and carotenoids, which were 12.43 ± 0.71 mg/kg of oil, and 1.35 ± 0.02 mg equivalent of β-carotene/g oil, respectively. In addition, the oils were analyzed by gas chromatography and their major fatty acids were linoleic, oleic, palmitic, and stearic. The ASO revealed the highest antioxidant activity in the quenching of 1,1-diphenyl-2-picrylhydrazyl (DPPH) with inhibition percentage of 89.2 ± 2.3 after 30 minutes of contact. Likewise, the oils were explored for the fortification of margarine. The physicochemical parameters of the formulated margarines comply with the standards. The Rancimat test showed that the highest induction time (16.54 h) was assigned to margarine enriched with 150 µg/g of WSO. Hence, this oil can has numerous applications in other food industries.

Keywords: vegetable oils / physicochemical parameters / antioxidant activity / margarine / oxidative stability

Résumé – Valorisation des sous-produits de l'abricot, du melon et de la pastèque par l'extraction des huiles végétales de leurs graines et la formulation de margarine. Les huiles végétales extraites des graines et des fruits oléagineux sont une source importante de composés bioactifs. Dans cette étude, les huiles de certains sous-produits de fruits ont été étudiées et leur composition et leurs propriétés ont été comparées. Les huiles de graines d'abricot (ASO), de melon (MSO) et de pastèque (WSO) ont été extraites par pression à froid. Les paramètres physico-chimiques et les teneurs en pigments ont été évalués selon des méthodes standards. Les valeurs des paramètres physico-chimiques ont révélé la pureté des huiles et il a été constaté que le WSO a les meilleures teneurs en chlorophylles et caroténoïdes qui étaient respectivement de $12{,}43 \pm 0{,}71$ mg/kg d'huile et $1{,}35 \pm 0{,}02$ mg équivalent β-carotène/g d'huile. De plus, les huiles ont été analysées par chromatographie en phase gazeuse et leurs principaux acides gras étaient les acides linoléique, oléique, palmitique et stéarique. L'ASO a révélé l'activité antioxydante la plus élevée dans la réduction du 1,1-diphényl-2-picrylhydrazyl (DPPH) avec un pourcentage d'inhibition de $89{,}2 \pm 2{,}3$ % après 30 minutes de contact. De même, les huiles ont été explorées pour l'enrichissement de la margarine. Les paramètres physico-chimiques des margarines formulées sont conformes aux normes. Le test Rancimat a montré que le temps d'induction le plus élevé

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(16,54 h) était attribué à la margarine enrichie par 150 μg/g de WSO. Par conséquent, cette huile peut avoir de nombreuses applications dans d'autres industries alimentaires.

Mots clés : huiles végétales / paramètres physicochimiques / activité antioxydante / margarine / stabilité oxydative

1 Introduction

Currently, the generation of food wastes is a critical difficulty, and their exploitation as food components for the elaboration of novel functional foods is necessary since customers request for foods that include ingredients with improved nutritional characteristics has augmented (Silva *et al.*, 2019). The usage of agri-industry wastes to conceive and elaborate novel foods with supplemented benefit is very critical for sustainability, considering this as resolution to diminish food losing, and ecological influence (Silva *et al.*, 2020).

Apricot (*Prunus armeniaca L.*) from Rosaceae family is prominent fruit since it is delightful and nutritive. Its seed (kernel) is a precious waste generated after consumption and processing (Al-Juhaimi *et al.*, 2021). Melon (*Cucumis melo L.*), and watermelon (*Citrullus lanatus L.*) from the Cucurbitaceae family are a famous fruits, consumed worldwide, that comprise considerable quantities of seeds (Petchsomrit *et al.*, 2020; Rabadán *et al.*, 2020).

The by-products of the selected fruits are an excellence source of oils with interesting composition and biological properties. Previous studies carried out on some of these oils confirmed the interest in use them as a possible functional ingredient (Siddeeg and Xia, 2015; Ramadan, 2019; Silva *et al.*, 2022).

Apricot seeds possess a considerable oil yield and this oil is rich in unsaturated fatty acids, sterols, tocochromanols and squalene (Ramadan, 2019). Additionally, ASO contains phenolic substances that play a favourable role in regards to the oxidative stability of oil and to the antioxidant capacity and health benefiting activities as cardiovascular diseases, cancer, tumors and ulcers (Ramadan, 2019; Al-Juhaimi *et al.*, 2021).

With regards with MSO, it have an appreciable quantity of sterols, tocopherols, and phospholipids which reinforce the relevance with its advantageous impact on the population where it assists in diminishing the hazard of cardiovascular pathologies by inhibiting the oxidation of polyunsaturated fatty acids (PUFA) (Ramadan, 2019).

As for WSO, it was stated as an excellent origin of essential fatty acids, carotenoids, tocopherols, thiamine, and phenolic compounds. Furthermore, WSO exhibited different biological properties mainly antioxidant, anti-inflammatory, cardioprotective and antimicrobial ones (Petchsomrit *et al.*, 2020).

Algeria possesses a broad diversity of flora, which can provide oils with various bioactivities. Interestingly, Algeria is among the top five apricot manufacturers worldwide (Ramadan, 2019). Nevertheless, there is few data on the composition and exploitation of the oil seeds.

Controlling oxidation is essential to manage the evolution of biological systems in their complexity, in particular in the case of foods whose degradation can have consequences for food safety. Among these food products, margarine, a plastic emulsion consisting essentially of two phases namely fatty and aqueous, also contains 2% of water and fat-soluble additives. Its composition is represented at 82% by a mixture of oils: the first target of oxidation (Fruehwirth *et al.*, 2021).

Lipid oxidation is a substantial reason of degradation of margarine over its fabrication that is mainly perceptible by the appearance development of disagreeable fragrance. This does not just denote oxidative deterioration products with possibly toxic features but conduct to the non-acceptance of the merchandises by customers (Himed and Barkat, 2014; Fruehwirth *et al.*, 2021). So, to ensure a long shelf life of dietary products such as margarine, natural antioxidants are widely employed. This study focuses beforehand on the comparison of the physico-chemical properties, the fatty acid composition, and bioactivity of ASO, MSO and WSO then on the impact of the substitution of the synthetic additive by these oils, as well as the determination of their capacity to protect margarines from accelerated peroxidation.

There are a number of papers communicating the fortification of margarine by natural products (Himed and Barkat, 2014; Chougui *et al.*, 2015; Kaanin-Boudraa *et al.*, 2021). However, the except of some reports dealed with the valorization of ASO, MSO and WSO by their use in other food products apart from margarine, papers describing the elaboration of functional foods using these oil by-products remain scarce. To the best of our knowledge, there are no references in the literature reporting the enrichment and preservation of margarine from oxidative deterioration using the chosen vegetable oils.

2 Material and methods

2.1 Chemicals and materials

All chemicals were acquired from Biochem Chemopharma (Montreal, Canada), Sigma-Aldrich (St. Louis, MO, USA) or VWR International GmbH (Vienna, Austria). All used solvents are of analytical grade.

Seeds of apricot (*Prunus armeniaca L.*) of variety "pêche de Nancy", of melon (*Cucumis melo L.*) of variety "inodorus", and of watermelon (*Citrullus lanatus L.*) of variety "grey bell" were used. These oilseed plants were grown in Northeast Algeria in the Bejaia department.

2.2 Oils extraction

Oils were extracted from sampled seeds by the cold pressing, which is achieved by directly pressing raw/dried seeds on a continuous screw press at low temperature. After 24-hour sedimentation, the extracted oil was separated from the sediment by decantation. Then, the oils were stored in dark glass bottles at a temperature of 4 °C, and then subjected to analyses (Brahmi *et al.*, 2020).

2.3 Determination of oil parameters

Physical characteristics of the oils were determined by measuring the refractive index using a refractometer RL 4 (PZO, Poland) at 40 °C. Absorbances in UV were measured by spectrophotometer (UV-Vis Spectrophotometer, spectro scan 50 Shimadzu, Kyoto, Japan) at 232 and 270 nm, respectively. Density of the oils was measured with respect to water density at a temperature of 20 °C.

Quality parameters (peroxide value (PV) and acid value (AV)) of cold-pressed oil were assessed according to the methods described by Duru *et al.* (2019).

2.4 Determination of chlorophyll and carotenoid contents

Measurement of the content of chlorophyll was performed according to AOCS method as described in our previous study (Brahmi *et al.*, 2020) and its content was expressed in mg of chlorophyll/kg of oil.

Carotenoid content in the oils was determined by using calibration curve of β-carotene (0.1–5 μg/mL) dissolved in *n*-hexane and recording the absorbance at 440 nm. *N*-Hexane was used for oil dilution to obtain the absorbances in a range of the calibration curve. The carotenoid contents were expressed in mg equivalent of β-carotene/g oil (Brahmi *et al.*, 2020).

2.5 Determination of the radical scavenging activity of the oils

Radical scavenging activity of vegetable oils was assessed by reduction of DPPH radical in toluene as reported by Ramadan and Moersel (2006). Toluenic solution of $DPPH^{\bullet}$ was freshly prepared at a concentration of 10^{-4} M.

For determination, 0.1 mg of each oil/mL toluene was mixed with 390 mL toluenic solution of $DPPH^{\bullet}$ and the mixture was vortexed for 10 s at ambient temperature (25 °C). Against a blank of pure toluene without DPPH, the decrease in absorption at 515 nm was measured after 1, 30 and 60 min of mixing using UV-Vis Spectrophotometer (spectro scan 50, Shimadzu, Kyoto, Japan). Antiradical action toward $DPPH^{\bullet}$ was estimated from the difference in absorbance with or without sample (control) and the percent of inhibition was calculated from the following equation:

$$\%\text{ of inhibition} = \left[\left(\text{absorbance}_{\text{control}} - \text{Absorbance}_{\text{test sample}}\right)/\text{Absorbance}_{\text{control}}\right] \times 100.$$

2.6 Determination of fatty acid composition of the oils

The analysis was carried out in the technical platform for physico-chemical analyzes of Bejaia, Algeria. The fatty acids were determined according to the method adopted by IOC with slight modifications. This method permits the determination of fatty acid methyl esters (FAMEs) from C12 to C24. In fact, the slight medications that we mentioned in this paper concern some operating conditions including the used gas carrier and detector temperature. In this regard, the gas carrier used in the IOC method is hydrogen although we used nitrogen, which is also an inert gas. In addition, the temperature of the FID detector was 270 °C although it was 250 °C in the IOC method. 0.1 g of the oil was purified through Silica-SPE cartridge then eluted with an admixture hexane: ether (50:50, *v/v*). The collected fraction was evaporated then dissolved in heptane. Triglycerides hydrolysis and transesterification, in basic medium, into fatty acids methyl esters (FAMEs) were performed by adding 0.5 mL of 2 N methanolic KOH. The upper part containing the FAMEs was recovered for GC injection. The fatty acids determination was performed with a Shimadzu-Nexis GC-2030 equipped with a flame ionization detector (FID). 1 μL of analytes were injected then separated on a high polar column Supelco SP-2380 (poly 90% biscyanopropyl–10% cyanopropylphenyl siloxane), 60 m length × 0.25 mm internal diameter × 0.20 mm film thickness (Sigma-Aldrich Co. LLC, St. Louis, MO, USA). A flow rate of 1.5 mL/min of nitrogen as a carrier gas and a split injection (50:1 split ratio) were applied. An oven program temperature had begun at 165 °C for 8 min then risen at 2 °C/min to reach a final temperature of 210 °C. Consequently, the run time was 30.5 min. The auxiliary gases were hydrogen at 32 mL/min, air at 200 mL/min, and nitrogen as a makeup gas at 24 mL/min. The identification was carried out using a standard chromatogram including FAMEs from C12 to C24 and using the chromatogram standard reported in the IOC referential method by comparing the obtained retention times (RT) to that reported in IOC. Each fatty acid was determined in percentage (%) represented by the area of the corresponding peak relative to the sum of the areas of all the peaks. The analysis was performed in triplicate for all samples from purification to injection.

2.7 Preparation of margarine samples

Oils at 50, 100, and 150 μg/g were employed to prepare three samples of margarine at CEVITAL agri-food industry (Bejaia, Algeria). A control margarine containing no oils but vitamin E was also formulated. After weighing the two phases and their ingredients, the mixture was poured into a stainless container where the emulsification took place with stirring for 20 min. At this point, the stability of the emulsion was incomplete; it had to undergo crystallization, which was carried out in a vessel containing ice-cold water. Stirring was carried out until a homogeneous margarine was obtained. The produced margarines were packaged in trays of 250 g each and stored at 6 °C.

After that, the margarines have been analyzed by determining humidity, pH, salt content, and melting point (Kaanin-Boudraa *et al.*, 2021).

2.7.1 Oxidative stability determined by Rancimat method

The oxidative stability of the margarines was evaluated by Rancimat method as reported in our previous work (Kaanin-Boudraa *et al.*, 2021). The oxidation induction time (OIT, in hours) was determined with the Rancimat apparatus (Metrohm 743, Herisau, Switzerland). For all analyses, 20 g of margarine were melted in an oven (DRY-Line®, VWR

Table 1. Physicochemical and quality parameters of the three studied oils.

	Apricot seed oil	Melon seed oil	Watermelon seed oil
Acidity (mg/g oil)	4.40 ± 0.21^a	0.80 ± 0.02^c	1.10 ± 0.01^b
Peroxide value (meq O_2/kg)	0.26 ± 0.01^a	0.20 ± 0.01^c	0.24 ± 0.01^b
Density	0.85 ± 0.00^a	0.78 ± 0.00^b	0.85 ± 0.00^a
Refractive index	1.4638 ± 0.0000^a	1.4666 ± 0.0000^a	1.4668 ± 0.0000^a
Absorbances in UV at 232 nm (K232)	0.29 ± 0.01^a	0.30 ± 0.01^a	0.30 ± 0.01^a
Absorbances in UV at 270 nm (K270)	0.0874 ± 0.0001^b	0.2901 ± 0.0001^a	0.2930 ± 0.0001^a

The results with different letters for each parameter indicate a statistically significant difference ($P < 0.05$, a > b).

International GmbH) at 80 °C for 25 min. Then, Rancimat vessels containing 3 g margarine were used for the analysis and an air rate of 10 L/h was applied. The OIT was evaluated using a temperature of 120 °C.

2.8 Statistical analysis

Results were presented as means ± standard deviation from three replicates of each experiment. A significance level $P < 0.05$ was employed to denote significant differences between mean values determined by the analysis of variance, post-hock Tukey tests (ANOVA) using Statistica 10 software.

3 Results and discussion

3.1 Physicochemical and quality parameters of the oils

The physicochemical and quality parameters of oils are illustrated in the Table 1. In addition to the characteristics listed in the table, the determination of the color of oils is a common and crucial factor for the production of margarine. Visual inspection of the extracted oils revealed that they had more or less dark yellow color; this indicates the occurrence of yellow pigments such as carotenoid compounds (Duru *et al.*, 2019), which can be beneficial for margarine fortification since they can act as natural antioxidants.

Peroxide value and free fatty acids are amongst the most prominent properties to assess the quality of edible oil samples. Acid value (AV) is an indication of liberated fatty acids by oil chemical breakdown but peroxide value (PV) is employed to measure the compounds generated from primary oxidation (Ok and Yilmaz, 2019). Indeed, the elevated AV is linked to the appearance of Free Fatty Acids (FFAs) that are more vulnerable to oxidation comparatively to the fatty acids found in the triacylglycerols (Rezig *et al.*, 2019).

Discrepancies were noticed among the studied oils in their acidity and peroxide values (Tab. 1). ASO had high value of acidity (4.40 ± 0.21 mg/g oil) followed by the WSO (1.10 ± 0.01 mg/g oil) and, MSO (0.80 ± 0.02 mg/g oil).

The extent of edibility of a fat is usually perceived to be conversely proportionate to the total level of FFAs. The low acidity of the investigated oils revealed that they are comestible and might have a prolonged storage life.

The PV of the investigated oils was in the range of 0.20–0.26 meq O_2/kg of oil. According to the Codex Alimentarius Commission, the peroxide value should not exceed 10 meq of peroxide oxygen/kg of oil.

The values revealed in this investigation are lower than those found in the literature for the ASO, MSO and WSO from different origins. PV of different apricot varieties from Pakistan ranged from 1.0 to 2.32 meq O_2/kg (Manzoor *et al.*, 2012). MSO and WSO are among the cucurbit seed oils, according to Mariod *et al.* (2009) these oils have PV, which varied from 2.3 to 4.1 meq O_2/kg. Melon grown in region of Plovdiv, southern Bulgaria provided seed oils with substantially elevated AV (1.5–2.1 mg KOH/g), however with bass PV (1.1–3.4 meq O_2/kg (Petkova and Antova, 2015). Seed oils extracted from *Cucumis melo L.* growing in west Algeria possess much higher acidity and peroxide values compared to those found in this present study which are respectively 4.01 mg KOH/g and 2.25 meq O_2/kg (Mulengi *et al.*, 2016). Oil from watermelon from Romania showed a moderate AV of 1.9 mg KOH/g and very high PV of 7.5 meq O_2/kg (Dumitru and Tutunea, 2017). AV and PV of seed oils extracted from some Cucurbitaceae including melon and watermelon ranged from 8.95 ± 0.84 to 9.60 ± 0.87 mg KOH/g oil and from 1.04 ± 0.11 to 9.56 ± 0.97 meq O_2/kg oil, respectively (Rezig *et al.*, 2019). In one study, the AV of the seed oils of melon and watermelon from Nigeria were high and respectively of 3.029 and 3.010 mg KOH/g, likewise the recorded peroxide indices were considerable with values of 26 and 27 meq O_2/kg, respectively (Duru *et al.*, 2019).

Nevertheless, kernel oil acquired from the seeds of some common sweet and bitter apricots from India were in perfect accordance with those reported in this study. The AV and PV varied from 0.2 (Maraghe) to 0.6 mg KOH per g of oil (Osku bitter), and 0.35 (Maraghe bitter) to 1.9 meq O_2 per kg of oil (Osku bitter), respectively (Shariatifar *et al.*, 2017).

Besides, the AV of WSO found in this current study is in the range of the contents of the oils obtained from the two varieties of Moroccan watermelon seeds using various extraction methods (1.40 ± 0.03 and 2.80 ± 0.06 mg KOH/g oil). Nevertheless, these authors observed greater PV (6.00 ± 0.08, 4.0 ± 0.67 and 3.80 ± 0.20 meq O_2/kg oil) (Ouassor *et al.*, 2020).

Vegetable oils density is conditioned by their composition in fatty acids, minor constituents and temperature (Neagu *et al.*, 2013). The density values recorded in this study were

0.85, 0.78, and 0.85 for ASO, MSO, and WSO, respectively. For ASO, the density value was lower than that established by Manzoor *et al.* (2012) which ranges between 0.87 and 0.93 mg/mL. The density of MSO is also lower compared to that found by Mulengi *et al.* (2016) which was 0.897.

The refractive index increases with unsaturation and the presence on fatty chains of secondary functions. So, a high refractive index allows concluding the occurrence of double bonds. In this current study, the measured refractive indexes were 1.4638, 1.4666, and 1.4668 for ASO, MSO and WMS, respectively.

Results of this study concur with the literature. By assessing the physico-chemical properties of seed oils extracted from various apricot varieties from Pakistan, Manzoor *et al.* (2012) recorded refractive index values ranged from 1.4655 to 1.4790. With a view to improve the effectiveness of oil extraction from wild apricot kernels from India by employing enzymes, refractive index fluctuate from 1.468 to 1.471 (Bisht *et al.*, 2015). Gayas *et al.* (2020) demonstrated that the refractive index was almost similar for all extraction methods studied for apricot kernels oil. It was 1.47 ± 0.00 using mechanical extraction, 1.45 ± 0.00 using Soxhlet and 1.46 ± 0.00 using ultrasound assisted.

The refractive indices and relative densities of the cucurbit seed oils ranged from 1.334–1.442 and 0.874–0.920 g/cm^3, respectively (Mariod *et al.*, 2009). Refractive index of MSO from Algeria was 1.470 (Mulengi *et al.*, 2016). The seeds oil of watermelon from Romania showed density of 945 and refraction index of 1.4731 (Dumitru and Tutunea, 2017). Refractive index value of the cold-pressed watermelon seed oil was 1.4696 (Ok and Yilmaz, 2019) and those extracted with *n*-hexane by Soxhlet has refractive index of 0.998 (Oragwu, 2020). The refractive indices were 1.46 and 1.47 Brix for MSO and WSO, respectively (Duru *et al.*, 2019).

Oxidation of unsaturated fatty acids leads to the formation of conjugated fatty acids, which could absorb UV light at 232 and 270 nm. Linoleic hydroperoxide can be measured at 232 nm and diketones and unsaturated ketones which can be quantified at 270 nm. So, the specific extinctions at 232 nm and 270 nm of oils can indicate their oxidation state (Loukou *et al.*, 2013). WSO possessed the highest K232 and K270 of 0.3 and 0.2930, respectively. The seed oil of the melon has an extinction of 0.30 at 232 nm and 0.2901 at 270 nm. For ASO, the K232 nm was 0.2942 and the K270 was 0.0874. These values are much lower than those of seed oils of Pakistan apricot varieties, which were 2.30–3.42 and 0.82–1.04, respectively (Manzoor *et al.*, 2012).

Several factors can have an impact on the parameters of oil as found by studies carried out on ASO, MSO and WMS. Kiralan *et al.* (2018) demonstrated that the storage period during 12 days under oxidation circumstances affects significantly the quality parameters of apricot kernel cold-pressed oils from Istanbul (Turkey). PV values of AKO achieved the highest value (54.5 meq O_2/kg) after 10 days of storage. Similarly, the maximum K232 (conjugated dienes) was attained (10.91).

In the study of Bisht *et al.* (2015), the usage of enzymes demonstrated that the different handling resulted in extracted oil from apricot kernels from India with AV ranging from 3.77 to 5.30 KOH/g.

The method of extraction adopted have also importance, the oil of seed apricot grown in Hebei Province (China) parameters were impacted by cold pressing, heat pressing, and refining of sun-dried and baked apricot kernels process. AV were 0.36–1.40 mg KOH/g, PV were 2.09–5.62 mmol O_2/kg, and absorbance values at 232 and 268 nm were of 0.70–0.85 and 0.20–0.38, respectively (Zhou *et al.*, 2016). Similarly, seed oil extracted from apricot growing in India country using ultrasound method showed AV, which rise from 2.27 to 2.69 mg KOH/g with temperature augmentation (Gayas *et al.*, 2017). Debitterizing caused also the rising of the PV and AV of the ASO and hence affected the oils quality by oxidative rancidity. AV varied from 1.43 ± 0.22 (blanched apricot kernels) to 2.22 ± 0.22 (debitterized kernels (60 °C, 6 h) mg KOH/g whereas PV from 3.37 ± 0.14 (blanched apricot kernels) to 4.57 ± 0.25 meq O_2/kg (debitterized kernels (50 °C, 8 h) (Song *et al.*, 2018).

The oil extracted from apricot seeds from India by hexane adopting different extraction methods showed an acidity of 2.71 ± 0.01 mg KOH/g oil by mechanical, 2.86 ± 0.03 mg KOH/g oil by Soxhlet and 2.73 ± 0.01 mg KOH/g oil by ultrasound assisted extraction. Regarding the PV, the oil obtained by mechanical extraction have the lowest PV (5.03 ± 0.02 meq O_2/kg oil), followed by oil from Soxhlet (5.09 ± 0.01 meq O_2/kg oil) but the oil extracted by ultrasound assisted technique recorded the highest PV (5.17 ± 0.05 meq O_2/kg oil) (Gayas *et al.*, 2020). Nevertheless, the oils extracted by SC-CO_2 and cold-pressed process from sweet apricot kernel seed cultivar from Croatia has the same PV (0.96–0.98 meq O_2/kg oil) (Pavlović *et al.*, 2018).

By investigating the impact of the extraction method on the oil extracted from golden melon seeds from Shanghai (China), the acid and peroxide amounts varied from 0.69–0.79 mg/g and 5.17–5.79 mmol/kg, respectively (Chen *et al.*, 2021).

In the same context, oils from melon of Tomelloso, Spain origin resulted from hydraulic press were of moderately improved quality with acidity of 0.30 ± 0.04% of oleic acid comparatively to oils resulted the screw press whose acidity was 0.41 ± 0.05% of oleic acid (Rabadán *et al.*, 2020).

The generated cold-pressed oils from seeds of watermelon from Mardin (Turkey), that were formerly processed with seed boiling and roasting exhibited almost the same acid value than those analyzed in this study (1.1 ± 0.0 and 1.2 ± 0.1 mg KOH/g oil for roasted and boiled oil seeds, respectively) (Ok and Yilmaz, 2019).

Solvent-extracted seed oil from watermelon of Uli (Nigeria) origin, displayed acidity and peroxide values of 30.80 mg NaOH/g oil, and 10 mg equivalent/g oil, respectively (Oragwu, 2020).

Regarding the cultivars, the extracted oils from five cultivars of apricot from Poland showed a PV that varied from 1.02 to 2.17 meq O_2/kg (Hargrand), refractive index from 1.4449 (Early Orange) to 1.4785 (Hargrand) and specific extinction values at 232 nm from 2.10 (Goldrich Sungiant) to 3.03 (Hargrand) (Stryjecka *et al.*, 2019).

3.2 Fatty acid composition of the oils

The prevalent fatty acids in all studied oils were oleic acid (C18:1 w9), linoleic acid (C18:2), palmitic acid (C16:0) and

Table 2. Fatty acid composition of apricot seed oil (ASO), melon seeds oils (MSO), and watermelon seeds oil (WSO).

Fatty acids	ASO	MSO	WSO	*P*-value (ANOVA)
Palmitic acid (C16:0)	4.82 ± 0.05^c	8.66 ± 0.03^b	9.60 ± 0.01^a	< 0.0001
Palmitoleic acid (C16:1 w9)	0.72 ± 0.01^c	0.10 ± 0.00^a	0.08 ± 0.00^b	< 0.0001
Margaric acid (C17:0)	0.05 ± 0.01^c	0.17 ± 0.03^a	0.08 ± 0.01^b	< 0.0001
Stearic acid (C18:0)	1.08 ± 0.03^c	5.59 ± 0.03^b	6.26 ± 0.01^a	< 0.0001
Oleic acid (C18:1 w9)	64.58 ± 0.27^a	24.06 ± 0.16^b	16.11 ± 0.02^c	< 0.0001
Vaccenic acid (C18:1 w7)	1.07 ± 0.04^a	0.65 ± 0.01^b	0.50 ± 0.01^c	< 0.0001
Linelaidic acid (C18:2)	27.29 ± 0.17^c	60.27 ± 0.18^b	66.84 ± 0.03^a	< 0.0001
Linolenic acid (C18:3)	0.10 ± 0.02^b	0.19 ± 0.04^a	0.17 ± 0.01^a	0.0139
Behenic acid (C22:0)	0.10 ± 0.00^c	0.22 ± 0.01^b	0.27 ± 0.00^a	< 0.0001
C18:1/C18:2	2.41 ± 0.02^a	0.41 ± 0.00^b	0.25 ± 0.00^a	< 0.0001
UFA/SFA	14.71 ± 1.01^a	5.76 ± 0.03^b	5.14 ± 0.01^c	< 0.0001

USA: unsaturated fatty acids; SFA: saturated fatty acids.
The results with different letters for each fatty acid contents indicate a statistically significant difference ($P < 0.05$, a > b).

stearic acid (C18:0). ASO showed the highest oleic acid content (64.58 ± 0.27%), followed by MSO (24.06 ± 0.16%), then WSO (16.11 ± 0.02%). Whereas, for linoleic acid (C18:2), WSO contains the highest content (66.84 ± 0.03%), followed by MSO (60.27 ± 0.18%) and ASO revealed an average concentration (27.29 ± 0.17%). The amounts of palmitic acid are not very important in the all oils; they are 9.60 ± 0.01%, 8.66 ± 0.03% and 4.82 ± 0.05 for WSO, MSO and ASO, respectively. It should be noted that ASO recorded a considerable UFA/SFA ratio (14.71 ± 1.01%) compared to two other oils (Tab. 2).

On the whole, results of this study concur with literature. The most abundant fatty acid in ASO was oleic (62.34–80.97%) followed by linoleic (13.13–30.33%), palmitic (3.35–5.93%), linolenic (0.73–1.03%) and stearic (1.10–1.68%) acids. Besides, the ASO are dominated by unsaturated fatty acids and polyunsaturated fatty acids (Manzoor *et al.*, 2012; Matthaus *et al.*, 2016; Dulf *et al.*, 2017; Juhaimi *et al.*, 2018; Kiralan *et al.*, 2018).

Moreover, ASO oil extracted by three techniques revealed a considerable content of unsaturated fatty acids (linoleic acid and oleic acid) and rather reduced amount of saturated fatty acids (Gayas *et al.*, 2020). Other authors demonstrated always the prevalence of the same fatty acids (palmitic, oleic and linoleic) in ASO (Shariatifar *et al.*, 2017; Jin *et al.*, 2018; Pavlović *et al.*, 2018; Song *et al.*, 2018; Stryjecka *et al.*, 2019).

The key fatty acids of MSO from West Algeria were linoleic (60.1%), oleic (25.3%), and palmitic (10.1%) acids (Mulengi *et al.*, 2016; Bouazzaoui and Mulengi, 2018). The main fatty acids of MSO cultivated in Tunisia (Mallek-Ayadi *et al.*, 2018) and Bulgaria (Petkova and Antova, 2015) were also linoleic and oleic acids. This oil offers also substantial contents of some SFA such as stearic (5–9%) acid (Silva *et al.*, 2019). The contents of these acids are in the range revealed by Kale *et al.* (2020) when analyzing the seed oils of 10 melon varieties: linoleic (57.1–74.7%), oleic (13.0–28.4%), and palmitic (7.0–10.1%) acids. Whereas, the amounts of the major SFA (palmitic acid, stearic acid, and butyric acid) in MSO varied from 1.09 to 11.9%, and the quantity of oleic acid was between 55.1 and 55.7% (Chen *et al.*, 2021).

Otherwise, the abundant fatty acids in Muskmelon oil seeds were hexadecanoic, heptadecanoic, octadecanoic, oleic acid, and pentadecanoic acids (Mehra *et al.*, 2015).

Similarly, in WSO oil, the predominant fatty acids were palmitic, stearic, oleic and linoleic acids (Dumitru and Tutunea, 2017; Ok and Yilmaz, 2019; Petchsomrit *et al.*, 2020). The same trend was observed by Ouassor *et al.* (2020) when studying two varieties of watermelon. According to Zarifikhosroshahi and Ergun (Zarifikhosroshahi and Ergun, 2021), the order of importance of fatty acid contents in WSO is as follows: palmitic, oleic, stearic, and linoleic acids. The main fatty acids were linoleic, oleic and palmitic acids for both varieties. However, myristic, linolenic and stearic acids were found in modest quantities. Linoleic, oleic, and palmitic acids in WSO were formerly mentioned to be extended from 45.1 to 76.2%, 0.33–33.66%, and 4.30–16.2%, respectively (Biswas *et al.*, 2017).

The MSO and WSO have a comparable composition of total saturated fatty acids, 20.24% and 24.01%, respectively which were represented by palmitic and stearic acids (Rezig *et al.*, 2019). Otherwise, Nigerian melon and watermelon seed oils contain pentadecanoic acid methyl ester (MSO: 4.91%; WSO: 4.43%), stearic acid (MSO: 3.64%; WSO: 41.77%), and methyl heptacosanoate (MSO: 39.16%; WSO: 1.32%). Concerning UFA, they were represented by 11-octadecenoic acid, methyl ester, (MSO: 46.05%; WSO: 46.28%) and oleic acid (MSO: 4.48%; WSO: 3.97%) (Duru and Maduka, 2021).

Numerous factors can influence the composition and fatty acid content of vegetable oils, among these conditions seed treatment. So, the fatty acid content of apricot kernel oil is dependent on the roasted procedure. Palmitic acid amounts varied from 4.38 (oven-roasted) to 4.76% (microwave roasted); oleic acid amounts were between 65.73% (oven-roasted) and 66.15% (control) and linoleic acid quantities ranged from 26.55 (control) to 27.12% (oven-roasted) (Al-Juhaimi *et al.*, 2021). The microwave power used for roasting apricot kernels affected also the composition of ASO's oils. The fatty acid profiling of kernel roasted at 360, 540 and 720 W were highly changed by roasting procedure comparatively to raw matter (Juhaimi *et al.*, 2018). The solid-state fermentation procedure has induced reductions of the palmitic and stearic

Table 3. Pigment content of the three oils studied.

	Apricot seed oil	Melon seed oil	Watermelon seed oil
Chlorophylls content (mg/kg) at 670 nm	0.10 ± 0.01^{c}	4.63 ± 0.04^{b}	12.43 ± 0.71^{a}
Carotenoids content (mg/β-carotene equivalent/g oil)	0.12 ± 0.01^{c}	0.41 ± 0.01^{b}	1.35 ± 0.02^{a}

The results with different letters for each pigment contents indicate a statistically significant difference ($P < 0.05$, a > b).

acids, and a considerable augmentation in the quantity of linoleic and oleic acids (Dulf *et al.*, 2017). Oleic acid quantities of ASO augment regarding maturation at the harvest periods but palmitic and linoleic acids amounts decline (Matthaus *et al.*, 2016).

The contents also depend on the variety, Kabaası variety showed the best content of palmitic acid (6.78%) then Çataloğlu variety (5.87%) (Juhaimi *et al.*, 2018). However, no significant changes were recorded according to the extraction methods (ultrasonic, mechanical and solvent) in ASO (Gayas *et al.*, 2020).

The fatty acid compositions of the MSO obtained by several techniques namely hot-pressing, cold pressing and ultrasound-assisted aqueous enzymatic extraction were distinct (Chen *et al.*, 2021). Oils resulted from the various melon cultivars reveled an elevated fluctuation in the concentrations of linoleic (51–69%) and oleic (15–34%) acids (Rabadán *et al.*, 2020).

The extraction method as well as the variety of watermelon showed an effect on the linoleic and oleic acids level. The best content of linoleic acid was attributed to the oil resulted from Soxhlet (67.43%) for the variety "lanatus". Otherwise, the best oleic acid level (19.08%) was attributed to the oil of variety "citroides" extracted by cold pressing (Ouassor *et al.*, 2020). The method of preservation of watermelon fruit (room temperature, at +4 °C for 12 months and newly harvested seeds) has shown an effect on the fatty acid contents of the oils of its seeds. The content of linoleic acid was elevated in samples stored at 4 °C. Besides, the ratio of polyunsaturated fatty acid to saturated fatty acids was determined as 1.16, 1.20, and 1.27 for the samples stored at room temperature, 4 °C, and newly harvested, respectively (Zarifikhosroshahi and Ergun, 2021).

3.3 Pigment content of the oils

Chlorophyll and carotenoid pigments are indicators of the quality of seed oils. According to obtained results, watermelon seeds oil gave the highest content of chlorophyll (12.43 ± 0.71 mg/kg oil) while the oil of melon seeds exhibited a moderate amount of 4.63 ± 0.04 mg/kg oil and apricot seeds oil recorded a very low level (0.10 ± 0.01 mg/kg). The same trend was noticed regarding carotenoid contents of the investigate oils. Watermelon seed oil contains the best concentration (1.35 ± 0.02 mg equivalent of β-carotene/g oil) followed by melon seeds oil (0.41 ± 0.01 mg equivalent of β-carotene/g oil), while apricot seeds oil was poor in this pigment (0.12 ± 0.01 mg equivalent of β-carotene/g oil) (Tab. 3).

Chlorophylls are linked to oxidative phenomena by their catalytic actions as pro-oxidants in the presence of light and antioxidants in the dark (Li *et al.*, 2019). Carotenoids are also involved in the mechanisms of auto-oxidation and photo-oxidation (Ouassor *et al.*, 2020). Indeed, carotenoids are very effective inhibitors of photoxidation induced by chlorophyll pigments (Grati Kammoun *et al.*, 1999).

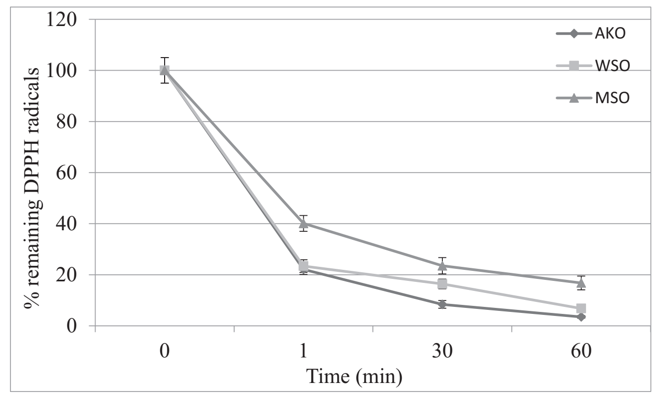

Fig. 1. Scavenging effect of vegetable oils during DPPH assay as measured by changes in absorbance at 515 nm. Error bars show the variations of three determinations in terms of SD. ASO: Apricot seed oil; MSO: melon seeds oils; WSO: watermelon seeds oil.

In the same line of our results, the carotenoids content of the kernels oil of 15 apricot genotypes from India were low and ranged from 1.5 to 5.3 mg/kg of oil (Gayas *et al.*, 2017).

β-carotene levels in seeds of five apricot cultivars grown in Poland ranged from 42.3 to 66.8 μg/g (Stryjecka *et al.*, 2019).

Górnas *et al.* (2017) established in their study that lutein, zeaxanthin, β-cryptoxanthin and β-carotene were the principal (76–94% of the total carotenoids) carotenoids in ASO, they also reported that the compositions are related to the genotypes.

The carotenoid content of the WSO depends on the variety and the method of extraction. Oil obtained by cold pressing from the variety var. citroides exhibited the best amount (73.95 ± 0.20 mg/kg), the oil resulted by Soxhlet from the variety lanatus comes second (24.78 ± 0.14 mg/kg) while the oil extracted using sonotrode ultrasound assisted extraction from citroides var showed the lowest content of 18.31 ± 0.22 mg/kg (Ouassor *et al.*, 2020).

3.4 Radical scavenging activity of the oils

The capacity to neutralize the DPPH$^{\bullet}$ by the three oils studied increases as a function of time (Fig. 1). It's obvious that ASO has higher anti-free radical activity, followed by WSO, and MSO gave low effect.

The anti-DPPH$^{\bullet}$ capacity of the oil extract of roasted apricot kernels varied between 2.55% (oven) and 19.34%

Table 4. Physico-chemical analysis of margarines formulated with the three oils.

	Control margarine	MASO	MMSO	MWSO	ISO662 standard (1998-90-1)
Moisture (%)	15.97 ± 0.01^a	15.30 ± 0.67^c	15.23 ± 0.74^d	15.87 ± 0.10^b	16
Salt content (%)	0.33 ± 0.01^c	0.37 ± 0.04^a	0.35 ± 0.02^b	0.37 ± 0.04^a	0.1–0.4% max
Melting point (°C)	35.00 ± 0.01^a	35.1 ± 0.1^a	35.4 ± 0.1^a	35.3 ± 0.3^a	34–37
pH	4.50 ± 0.01^b	4.80 ± 0.03^a	4.30 ± 0.02^c	4.10 ± 0.01^d	4–5.5

MAKO: margarine formulated with apricot seed oil; MMSO: margarine formulated with melon seed oil; MWSO: margarine formulated with watermelon seed oil.
The results with different letters for each parameter indicate a statistically significant difference ($P < 0.05$, a > b).

(microwave-roasted). Additionally, radical scavenging effect increased with roasting (Al-Juhaimi *et al.*, 2021).

The anti-DPPH$^\bullet$ activity of the melon seeds oil from Sudan was good with IC_{50} of 25.25 mg/mL (Azhari *et al.*, 2014).

WSO at 1 g/mL from Romania exhibited a moderate antiradical activity (46%) (Dumitru and Tutunea, 2017). Ouassor *et al.* (2020) assessed the antioxidant activity by the free radical DPPH of the methanol fraction of different WSO. They demonstrated that the technique of obtaining the oil and the variety of watermelon considered have a great impact on the activity. The results found oscillate between 51.1 ± 0.1% for the cold-pressed oil sample of var. lanatus and 84.8 ± 0.04% for Soxhlet var. citroides.

Watermelon seeds oil extract from Thailand exert a potent ability to scavenge DPPH$^\bullet$ with 0.894 mg α-tocopherol equivalent/g dried seeds ($IC_{50} = 3653.29 \pm 539.31$ mg/mL) (Petchsomrit *et al.*, 2020).

3.5 Physico-chemical analysis of the formulated margarines

Given the interesting characteristics of the oils explored, they are used to enrich table margarine. The results of the physico-chemical analysis of the formulated margarines as compared to the control are depicted in Table 4.

We noticed that there is no difference between the moisture content of the control margarine (M0) and the three other margarines elaborated (MASO, MMSO, and MWSO) with the vegetable oils studied, the levels were 15.97 ± 0.01, 15.30 ± 0.67, 15.23 ± 0.74, and 15.87 ± 0.10%, respectively. This is compatible with the initial formulation of the margarine, which is constituted of 82–84% of fatty phase and 16–18% of the aqueous phase. The addition of salt to margarine is intended to improve its organoleptic characteristics and also to inhibit the growth of certain bacteria, which prolongs its shelf life. In the present study, the salt content (NaCl) of the three margarines produced were 0.37 ± 0.04, 0.35 ± 0.02 and 0.37 ± 0.04%, respectively. The control margarine had a salt amount of 0.33 ± 0.01%.

The melting point of the formulated margarines were 35.4 ± 0.1, 35.3 ± 0.3 and, 35.1 ± 0.1 °C for MMSO, MWSO and MASO without significant difference with M0 (35 °C). This characteristic is related to the fatty acid composition, long chain saturated fatty acids have a higher melting point than the short chain unsaturated fatty acids.

As regarding the pH values, which provides information on the state of freshness of the sample, they varied between 4.10 and 4.80. This means that the amounts of acetic acid added to margarines were respected. The pH, between 4 to 5.5, is intended to make margarine taste good and prevent microbial growth (Karleskind and Wolff, 1992).

3.6 Oxidative stability of the formulated margarines

The association of considerable oleic and linoleic acid amount in oil is of benefit, as they are regarded stable and nutritious oils, which aid avoids different pathologies (Gayas *et al.*, 2020).

Considering that all the investigated oils were a substantial source of both oleic and linoleic acids, they might be employed as cooking or salad oils or might alike be employed for margarine formulation (Rezig *et al.*, 2019).

Oils are very important in human diet because of the high contents of essential fatty acids and antioxidants. They may be a good substitute for synthetic products for food applications.

Margarine is an emulsion prone to oxidation, and it is well known that vegetable oils are rich in antioxidants; their addition during margarine formulation could have an advantageous impact.

According to the results of this study (Fig. 2), the induction times of the three formulated margarines with 150 μg/g of ASO, WSO and MSO were 15.36 h, 16.54 h and 14.66 h, respectively; these results prove the oxidative stability generated by the investigated oils. Nevertheless, commercial margarine containing vitamin E gave the best induction time of 19.47 h. The margarine enriched with WSO has a higher induction time, followed by a margarine prepared with ASO but the induction time given by a margarine prepared with MSO was the lowest.

These oils can improve antioxidant capacity by preventing oxidation of PUFA thanks to their active components such as carotenoids and phenolics.

Unfortunately, there was no study in the literature for comparison. The oils studied have instead been applied in the production of other foods. As application of the apricot kernels oil, Saini *et al.* (2021) reported in their review study its use in macaroon paste and for enrichment of noodles. MSO has been mixed with peanut oil and the findings revealed that this can help to enhance some properties of the oil (nutritional and functional) (Siddeeg and Xia, 2015).

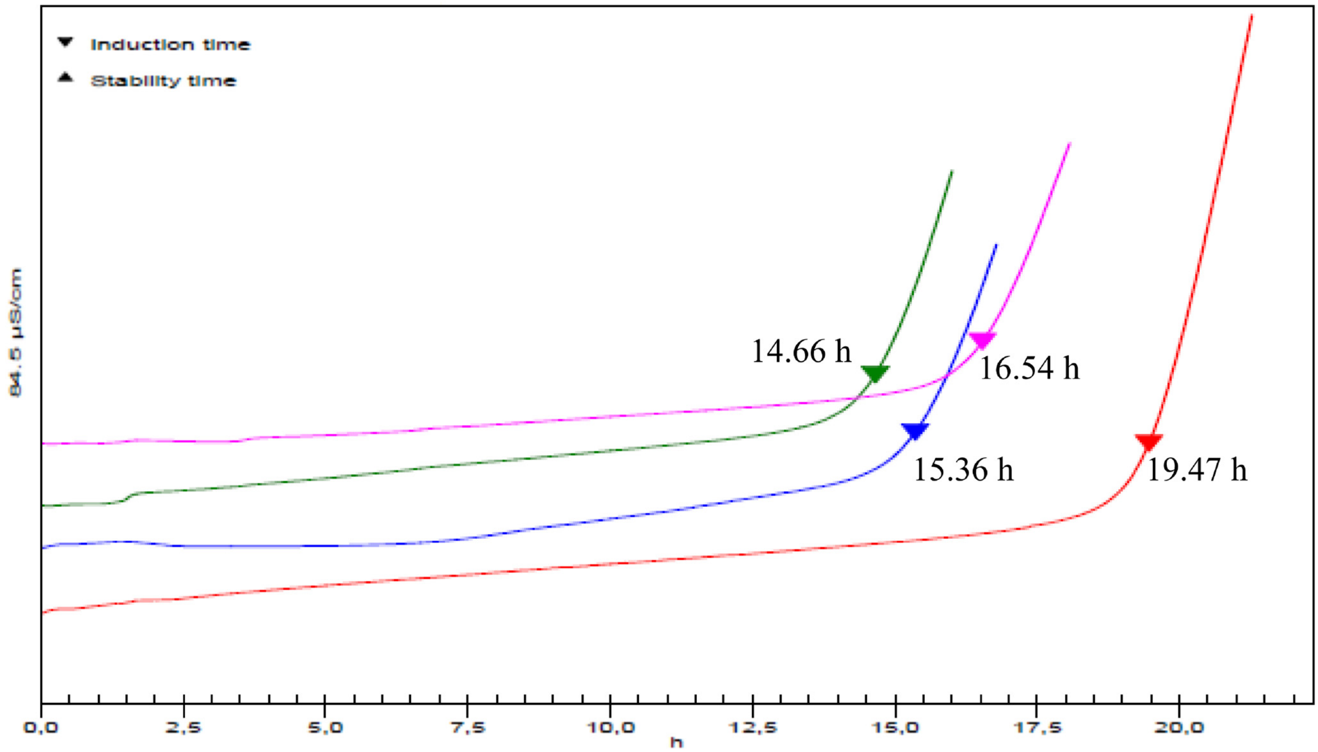

Fig. 2. Stability of margarines formulated with apricot seed oils (ASO), melon seed oils (MSO), and watermelon seeds oil (WSO) to accelerated oxidation. Red line: control margarine (marketed); blue line: margarine formulated with 150 ppm ASO; green line: margarine formulated with 150 ppm MSO; pink line: margarine formulated with 150 ppm WSO.

4 Conclusion

The seeds of apricot, melon, and watermelon were valued in this study by the extraction of their oils and their use in protecting a food product from oxidation, which is margarine.

The oils analysis showed that their physicochemical and quality parameters were are up to standard and of satisfactory quality. Furthermore, according to GC-FID analysis of these oils, oleic (C18:1 w9), linoleic (C18:2), palmitic (C16:0) and stearic acids (C18:0) were the main fatty acids in the three oils. It should also be noted that the oils studied exerted a significant scavenging effect with respect to the $DPPH^{\bullet}$. Oils at 150 µg/g added to table margarine have helped in protecting it from oxidation and watermelon seed oil showed better effect without affecting the properties and quality of the margarine.

The oils studied can be used to improve the nutritional quality of margarine due to their richness in fatty acids and to protect it from oxidation because they are a natural source of antioxidants.

Conflict of interest

The authors declare that they have no conflicts of interest in relation to this article.

Authors contributions statement

Fatiha Brahmi: Conceptualization, Investigation, Methodology, Writing–Original Draft. Boualem Chennit: Methodology, Data analysis, Writing–Review and Editing. Houria Batrouni: Methodology. Kenza Benallaoua: Methodology. Khodir Madani: Supervision. Lila Makhlouf-Boulekbache: Conceptualization, Supervision.

All authors have read and agreed to the published version of the manuscript.

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