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Compound Identification and Physicochemical Properties of Syrian Plum Molasses

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Abstract

The present work describes the evaluation of plum molasses (PM) as typical Syrian products called "Dibis Al-Khookh". Some nutritional, microbial, chemical, and physical properties and storage stability of PM were investigated. The PM contents of moisture, crude protein, crude fat, ash, total sugar, and reducing sugar were: 53.31, 3.22, 0.40, 3.63, 37.44, and 28.50 %, respectively. The results confirmed that PM gives considerable amount of human mineral requirement. The total acidity (TA), pH value, total volatile basic nitrogen (TVBN), total soluble solid (TSS) and viscosity were 3.76%, 3.16, 27.57 (ppm), 37.05 °Bx and 19.33 mP.Sc, respectively. We observed that the PM was free from microorganisms. Since microorganism's growth is influenced by pH of a medium, it is logical to suggest that PM could be stored longer as low pH has the ability to inhibit microorganism's growth. PM was shown in this study to be a nutritional food and food ingredient. Indeed, PM could be also exploited in food industry and used as natural ingredient or additive.

Keywords

Syria, Plum molasses, Dibis, Phsico-chemical properties

Introduction

The consumption of fruits and vegetables has been linked to health benefits, such as protection against diseases associated with oxidative stress, for example, chronic diseases [1].

Plum (*Prunus* Sp. L.) belongs to Rosaceae family is widely distributed in Asia [2]. The genus *Prunus* includes about 200 species that are widespread in the temperate climate, particularly in East Asia [3]. Prunus species are rich in phytochemicals and biologically active ingredients [4]. Biochemical compounds in plums have been reported to have beneficial effects on the human body [5]. *Prunus* contains many important species who's usually consumed fresh or processed; however, they are also used in food industry for production of jams, beverages: liquor, wine, juice, compote and tea [6].

The fruits of wild plum (P. *Spinosa*) are commercially unexplored due to their astringent taste, therefore, the processing of fruits into products such as juices, purees, jams, canned fruits, and marmalades can be an alternative to prolong the accessibility and the consumption of fruits [7].

Molasses or 'Dibis' is a traditional food well known in many Mediterranean countries including Tunisia, Lebanon, Syria and Turkey [8, 9]. Molasses is a kind of concentrated fruit juice, and a naturally nutritious ingredient with high percentage of sugars and minerals, so it is used as an alternative to jam, sugar or honey [10]. In addition, it is a good source of phyto-chemical compounds with health promoting properties such as antioxidant, antimicrobial and anticancer activities [11].

The Syrian diets contain many foods, among which the molasses, are believed to have antioxidant effects but without much scientific evidence. Plum molasses (PM), a concentrated plum juice, may be rich in more efficient antioxidants than that found in the juice. No research has been done on this product so far. The objective of this study is to fill this gap and determine: 1) the proximate composition (moisture, crude protein, crude fat, ash total sugar, reducing sugar, and minerals); 2) the microbial load (total viable counts (TVC), total coliform counts (TCC) and total mould and yeast (TMY)); 3) the physic-chemical properties (total acidity (TA), pH value, total volatile basic nitrogen (TVBN), total solids solid (TSS), viscosity and color).

Materials and Methods

Treatments and analysis performed

We used plum molasses (PM) known as Dibis Al-Khookh, made from a wild plum variety grown in *Brood regon*, Damascus, Syria. The investigated samples were produced according to a traditional process which consisted of peeling the fruits, dispersing the grains, and pressing them manually to have a juice. The juice is boiled for more than six hours to obtain a concentrated substance called "molasses' or 'Didis". Then PM were weighed as in the sampling plan and transferred into polyethylene bottle, each bottle of PM (0,5 kg) was considered as a replicate. The sample used was a PM obtained from a local manufactory. No changes were made to the way in which the PM are usually prepared and packaged in this industry. PM was stored for 12 months in dark glass bottles at ambient temperature (18-25 °C) under relative humidity (RH) of 50-70%. Color measurement, chemical compositions, viscosity measurement and microbiological of PM samples were performed at beginning of the study and after 6 and 12 months of storage.

Chemical analysis

Chemical composition: Standard procedures of the Association of Official Analytical Chemists (AOAC) were used for determinations of moisture, crude protein, crude fat, total sugar, reducing sugars and ash contents [12].

Determination of minerals: Mineral content including: major elements (Potassium (K), Magnesium (Mg), Chlorine (Cl), Sodium (Na) and Calcium (Ca)); trace elements (Aluminum (Al), Bromine (Br), Cobalt (Co), Iron (Fe), Manganese (Mn), Rubidium (Rb), Zinc (Zn), Barium (Ba), cupper (Cu) and Cerium (Ce)); and toxic elements (Chromium (Cr), Arsenic (As), Lanthanum (La), Selenium (Se), Strontium (Sr), and Vanadium (V)) were determined in PM samples using Instrumental Neutron Activation Analysis (INAA) according to the standards methods descript by Al-Bachir et al. [13].

Physicochemical properties determination

Colors of PM samples were analyzed by measuring CIE L* (brightness; 100: white, 0: black), a* (+: red; -: green) and b* (+: yellow; -: blue) and the color difference (ΔE*) values parameters with AvaSpec Spectrometer Version 1, 2 June 2003 (Avantes, Holland) at wave length of 670 nm [14].

Total Soluble Solids TSS (°Brix) of PM was directly measured by hand refractometer, previously calibrated with distilled water, range of 0-100 °Brix at 20 °C. The pH values were determined using an HI 8521 pH meter (Hanna Instruments, Woonsocket, RI, USA). Titrable acidity (TA) was determined as % citric acid by titration with 0.1N from NaOH as described by [12]. Total volatile basic nitrogen (VBA) in the sample in terms of mg VBN/kg PM (ppm) was determined [15].

Viscosity measurement: The viscosity of the PM was measured with viscosity meter Model (RTM) Townson + Mercer 061 928 6211, using U column (Instrument No. 5769) and fixed No. 0.027171, and expressed as mPa. s [14].

Microbiological analysis

Microbiological analyzes were performed on PM product according to the method proposed by AOAC [12]. The main microorganisms searched were: Total viable counts (TVC) using plat count agar (PCA) (Oxoid, CM 325, UK) (30 °C, 48 h). Total coliform counts (TCC) using Violet Red Bile Agar (VRBA) (Oxoid, CM 485, UK) (37 °C, 48 h). Total mould and yeast (TMY) using Dichloran Rose-Bengal Chloramphenicol Agar (DRBC) (Merck, 1.00466, Germany) (25 °C, 5 days).

Statistical analysis

All experiments were replicated three times. Data were expressed in the form of means ± standard deviation (SD). The data were subjected to the analysis of variance (ANOVA). The difference was of statistical significance at $p < 0.05$ between means using the commercial statistical package (SPSS, Inc, Chicago, IL, USA).

Results and Discussion

Chemical composition of PM

The proximate analysis is a set of methods to get information about the nutritional value of food. As far as we know, this study describes for the first time the quality of Syrian PM. Therefore, the results were mainly compared to those reported for other fruits molasses or jam. The proximate component of the PM (Dibis Al-Khookh) obtained in proximate analysis are given as a percentage of fresh basis in the table 1. The contents of moisture, crude protein, crude fat, ash, total sugar, and reducing sugar were: 53.31 ± 0.52, 3.22 ± 0.82, 0.40 ± 0.02, 3.63 ± 0.03, 37.44 ± 1.38, and 28.50 ± 0.18%, respectively. Yılmaz et al. [16] have found the moisture content of the pomegranate molasses 24.4%. The high content of total sugar (37.44%) makes this concentrated PM easy to process both by fermenting processes (alcoholic fermentation), for concentrated products (paste, jam) [17]. The presence of reducing sugars (28.50%) naturally in PM could provide a good source of rapid energy since they pass easily into the blood without digestion [8]. With the increase in initial sugar level, reducing sugars content were increased. Reducing sugar may affect to the quality and aroma characteristics of PM. According to Asikin et al. [18] this involved in Maillard reaction that produce brownish color and baked-like aroma.

Table 1: Influence of storage time on proximate composition of Syrian plum molasses.

P-level	12	6	0	Storage period /(Months)
		Characteristics		
**	±56.720.25[b]	0.09±57.45[a]	[c]0.52±55.31	**Moisture (%)**
**	[a]0.47±6.36	[b]0.08±4.60	[c]0.82±3.22	**Crude protein (%)**
NS	[a]0.07±0.45	[a]0.02±0.40	[a]0.02±0.40	**Crude fat (%)**
**	[a]0.09±3.81	[b]0.18±2.60	[a]0.03±3.63	**Ash (%)**
**	[c]0.86±32.66	[b]0.18±34.95	[a]1.38±37.44	**Total sugar (%)**
**	[b]0.40±27.37	[c]0.10±25.48	[a]0.18±28.50	**Reducing sugar (%)**

Different superscript uppercase letters show differences between samples (P < 0.05).
NS: not significant.
** Significant at p<0.01.

With respect to total sugars and storage (Table 2) there was a significant (P ≤ 0.05) decrease up to 6 months (37.44%), with more and significant (P ≤ 0.05) increase at the end of storage (32.55%), a behavior that can be associated with increased crude protein and humidity during storage [19].

Table 2 : Elements concentrations (µg/g) of Syrian plum molasses.

	Concentrations (µg/g)
Major elements	
K	13350±472
Mg	766.6±91.7
Cl	2999±0670
Na	19495±236
Trace elements	
Al	1408±29
Br	10.65±0.21
Co	0.061±0.006
Fe	16.44±1.23
Mn	5.84±0.52
Rb	4.33±0.13
Sb	0.0107±0.0046
Zn	9.54±0.59
I	2.16±0.68
Toxic elements	
Cr	1.31±0.07

Ash content is an index to the nutritive value of foods. The investigated PM was also found to contain minerals as ash in the range of 3.63%. This result seems to be in close proximity to those reported by other researchers for carob molasses 2.97% [9].

The crude protein content of the PM (Table 1) was 3.22 ± 0.82%. According to previous research, crude protein content of papaya jam was 3.15% [20]. Crude protein values of avocado, pineapple, papaya, passion fruits, watermelon varied from 4.4 ± 1.0 to 23.3 ± 0.5% dry basis [21].

The crude fat content of the PM was 0.40 ± 0.02. The values obtained are within the limits provided by several authors who point out that the fat content in the fruits of plums is 0.1 - 0.4% [22]. Previous research states that different parts of fruits presented total lipids values in the range of 1.1 ± 0.2 to 28.7 ± 7.9% dry bases [21]. Generally, the fat content of fruits and vegetable is low [23].

The results of this study indicated that the PM contains very low levels of proteins and devoid of crud fat. Whereas fat content of PM did not change during storage due to its too low-fat content (0.4%) (Tables 1). These results might be due to the metabolic activities and hydrolyzing enzyme activity in stored PM [24].

Similar nutritional composition was reported for molasses produced from others fruits such as date [11], juniper [25], and carob molasses [9].

The neutron activation analysis (NAA) technique allowed us to obtain the concentration of 14 elements in PM (Dibis Al-Khookh). As shown in table 2, the mineral composition of the PM samples was dominated by Chlorine (Cl) (29990 µg/g), followed by Sodium (Na) (19495 µg/g), potassium (K) (133350 µg/ g), Aluminum (Al) (1408 µg/g), and Magnesium (Mg) (766 µg/g). Considering the micronutrients, Iron (Fe) (16.44 µg/g), Bromine (Br) (10.65, µg/g), Zinc (Zn) (9.54 µg/g), Manganese (Mn) (5.84 µg/g), Rubidium (Rb) (4.33 µg/g), iodine (I) (2.16 µg/g), and Chromium (Cr) (1.31 µg/g) were the most abundant elements. While Cobalt (Co), and Sb were present at low level in the range of 0.061 and 0.011 µg/g, respectively. According to this experiment, it is confirmed that PM "Dibis Al-Khookh" gives considerable amount of human mineral requirement. Previous studies Tounsi et al. [8] conducted on the mineral profile of carob molasses have also stated that the mineral composition of the samples was dominated by K (~ 1900 mg/100 g) followed by Ca and Mg in the range of 360 and 270 mg/100 g, respectively. Considering the micronutrients, Fe, and Zn were the most abundant elements (~ 3 and ~ 2 mg/100 g, respectively), while Cu and Mn were present at low level in the range of 0.1–0.3 mg/100 g. On other hand Tetik et al. [26] reported that the mineral profile of carob Pekmez have also stated K as the major element, in addition to the other macro elements (Na, P, Ca and Mg) at different concentrations, and the microelements

(Cu, Fe, Zn and Mn) at very low concentrations. İncedayi et al. [27] revealed that the pomegranate molasses samples were rich in minerals, especially in K (450 - 4700 mg/100g), Ca (71.88 - 1803.63 mg/100g), Mg (7.48 - 409.10 mg/100g) and Fe (1.05 - 22.99 mg/100g).

These differences showed between mineral concentrations may arise from the differences between mineral composition of fruits used as raw materials for molasses production, and processing steps applied such as clarification and filtration. Leterme et al. [28] had reported that several factors like variety, state of ripeness, soil type, soil condition, and irrigation system may cause variation in the mineral contents in different types of fruits as well as within different parts of the same fruit.

According to this experiment, it is confirmed that PM gives considerable amount of human mineral requirement and toxic elements does not contain within PM.

Minerals play a key role in different physiological functions of the human body, especially in the building and regulation processes. Fruits and its products are considered as a good source of dietary minerals. According to United State Department of Agriculture (USDA) the daily recommended intake (RDA) is: 1,000-1,200, 310-420, 4,700, 1,200-1,500, 0.9, 8-14, 1.8-2.3 and 8-11 mg per day of Ca, Mg, K, Na, Cu, Fe, Mn and Zn, respectively for Na and K intake is not more than 2.400 mg and 4700 mg respectively per day [23].

Chemical and physical properties of the PM

Physical and chemical characteristics of PM are given in table 4. The total acidity (TA), pH value, total volatile basic nitrogen (TVBN), total soluble solid (TSS) and viscosity were 3.76%, 3.16, 27.57, 37.05 °Bx and 19.33 mP.Sc, respectively.

Color changes of PM were monitored on the L*, a* and b* scales during storage time, colour differences (ΔE) were also calculated. As shows in table 1, color estimation of PM revealed L* (lightness) value of 38.59, a* (redness) value of 58.11, b* (yellowness) value of -15.14, and ΔE value 46.98. L*, a*, and b* values revealed that the PM used in this study was rich in pigments. The studied PM had high brightness (L*) (38.59) and redness (a*) (58.59) values which indicate a moderate quality product. A high redness (a*) value means excessive browning reactions and caramelization of sugar at high temperatures and Maillard reaction [9]. However, a high redness values a* are not desired

Lightness of the jams showed an increase in the first 6 months and a decrease until the end of the storage period. Color characteristics like brightness, redness, yellowness, and shine are among characteristics that can trigger consumer willingness at first glance [29].

Changes on the red hue of samples are inconsistent, as a result, no marked decrease was observed in the global a* values at the end of the observation period, the initial differences having been equalized. Results of the repeated measures ANOVA test of a* showed that the storage time did not significantly affect the results (p = 0.05). Some other authors found that the intensity of red hue was higher in cherry jams after storage [30].

Results of the repeated measures ANOVA test of b* values showed that storage time had a significant effect on the yellow-blue scale of the samples at $p<0.05$ significant level, according to the within-subject effect test. The effect of the storage time showed that samples stored for 6 and 12 months showed significantly lower b* values than the not stored samples.

ΔE values showed that the global color differences compared to the jams are higher at the end of the storage period. Color is an important quality factor for food, mainly for fruit-based products, such as jams, and other preparations. Therefore, the colour of the products should not change during storage [1].

The color formation in PM during storage could be attributed to both non-enzymatic browning and phospholipids degradation during storage [31]. The non-enzymatic browning is favored by heat and storage and includes a wide number of reactions such as Millard reaction, caramelization and chemical oxidation of phenols [32]. For instance, in the case of Millard reaction, the PM contains the required reaction, sugars and amine groups as found in protein molecules, to give the Millard reaction products [31].

The TSS is a refractometric index that indicates the proportion (°Brix) of dissolved solids in a solution [33]. The TSS of PM was 37.5±1.86° Brix at zero time. The high TSS in PM might have been due to richness of PM in the sugars. Research has shown that sugar content is positively correlated with TSS in fruit and in its products [34]. The °Brix values of molasses were mainly due to their soluble sugar's contents, mainly sucrose glucose, and fructose [9].

No available data in the literature about the °Brix values of PM, whereas a previous study reported similar values (42.57)

Table 3: Influence of storage time on proximate composition of Syrian plum molasses.

P-level	12	6	0	Storage period /(Months)
**	^{b}0.58±40.29	^{a}1.31±41.98	^{c}0.21±38.59	**L(Lightness)**
NS	^{a}0.97±58.06	^{a}2.21±58.00	^{a}0.60±58.11	**a (redness/greenness)**
**	^{b}1.42±18.68-	^{a}2.19±22.21-	^{c}1.45±15.14-	**b (yellowness/blueness)**
**	^{b}0.12±43.71	^{c}0.69±40.44	^{a}0.82±46.98	**ΔE (Total color difference)**

Different superscript uppercase letters show differences between samples (P < 0.05).
NS: not significant.
** Significant at p<0.01.

Table 4: Effect of storage period on total acidity (% Lactic acid), PH value, volatile basic nitrogen (VBN)(P.P.M), total soluble solid (°Brix) and viscosity (mP. Sc) of Syrian plum molasses.

P-level	12	6	0	Storage period /(Months)
			Characteristics	
**	^{b}0.12±3.37	^{b}0.05±3.38	^{a}0.03±3.76	**Total acidity (% Lactic acid)**
**	^{b}0.00±3.49	^{a}0.03±4.01	^{c}0.04±3.16	**PH value**
**	^{a}2.57±43.91	^{a}2.43±37.60	^{b}4.37±27.57	**Volatile basic nitrogen (ppm)**
**	^{a}0.26±37.95	^{b}2.15±31.68	^{a}1.86±37.05	**Total soluble solid (°Brix)**
NS	^{ba}0.00±19.00	^{b}1.00±18.00	^{a}0.58±19.33	**Viscosity (mP.Sc)**

Different superscript uppercase letters show differences between samples (P < 0.05).
NS: not significant.
** Significant at p<0.01.

for sugar free traditional plum jam [35]. The result of their study was a little different from the results obtained in this study, which is due to the difference in variety and composition of black plum that they used. In other study Ajenifujah-Solebo and Aina, [36] reported soluble dry matter = 68% for black plum (Vitex doniana) jam. Pinandoyo and Masnar [20] reported that the TSS of papaya jam was 68.5%. The wide variation in TSS is also apparently related to the difference in fermentability behaviour of the musts [37].

No significant differences were recorded between the samples at zero time and after storing for 12 months. There was a slight decrease up to 6 months (31.68o Brix) with a subsequent slight increase of around 37.95o Brix after 12 months storage in temperature of 25°C. Rababah et al. [38] evaluated the cherry jam between 0 and 15 days and found soluble solids values 11.25% to 66.30%.

The TA and pH value are a necessary factor in the maintenance of the product and food [39]. As shown in table 3 the TA (3.76%) and pH value (3.16) of PM were found to be inversely related with each other. The increase of pH value also indicated by reducing in acidity level [40]. No available data in the literature about the TA and pH value of PM. Whereas, several studies determined the pH value and acidity of other kind of molasses (commercial carob molasses) and reported different values in the range of 4.96–5.44 and 0.6–1.34% as citric acid, respectively [9, 27]. Mohammadi-Moghaddam et al. [41] showed that the acidity and pH value of the Black plum peel jam samples were in the range of 0.38–0.58% and 2.80–4.00, respectively. Ajenifujah-Solebo and Aina [36] reported pH = 3.42 and acidity = 0.34 for black plum (Vitex doniana) jam. In other study Culetu et al. [35] reported pH = 3.39–4.05, acidity = 0.84–2.32, for sugar free traditional plum jam. Also, Pinandoyo and Masnar, [20] reported pH = 3.27, acidity = 0.53, for papaya jam.

It was observed that the TA of PM decreased from 3.76 to 3.38% during the storage period of 6 months, with a further slight reduction at 12 months. Brandão, et al. [42] analyzing mixed fruit jam observed that during storage there was pH stability, a result like that of the present work. On other hand, Damiani et al. [19] found that the pH value of marolo and strawberry guava jam, during the first 6 months of storage, presented a slight decrease from 3.31 to 3.27 and after 12 months the pH value increased to 3.33. Similar results reported by Mesquita, et al. [43].

TA gives a measure of the amount of acid present in a fruit or fruit products. The decline in acidity during storage from 3.76% to 3.37% may be attribute to the starch hydrolysis leading to an increase in total sugars and a reduction in acidity [44]. The change in pH values could be attributed to the chemical reactions like apparition of Maillard reaction products [45].

In general, molasses was characterized by acidic pH which could be explained by either degradation of sugars into acids [46]. The nature and concentration of the organic acids found in fruit products are of interest because of their important influence on the organoleptic properties and stability of fruit products [47].

The results of the present study show that, the overall observed value of TVBN was in between 27.57 and 43.91 ppm. However, there is no information about the TVBN or aroma profile of PM. Therefore, the present study presents the first report that describes the volatile profile of Syrian PM. The content of some volatile compounds increased during storage. However, the TVBN is related to protein degradation, and the observed increases may be attributed to the formation of ammonia or other basic compounds due to microbial activity [48]. According to Koppel et al. [49] decreased volatile concentration could be linked to less intense flavour attributes in the fruit products

Viscosity is considered as an important physical property related to the quality of liquid food products. The viscosity values of the investigated PM were 19.33 mPa s. A previous study carried out in rheological characteristics of carob Pekmez revealed higher viscosity values (~ 800 mPa s at 30 °C) [10]. The viscosity values of the carob molasses were found to be 1182 – 1284 mPa s [9]. Guilherme et al. [50] evaluated the quality of molasses prepared from mesquite pod and cashew apple and mentioned that the syrups viscosity was lower than those reported for honeys (4500–3600 mPas). Viscosities of the pomegranate molasses samples were found between 200 and 1800 mPa.s [27]. The difference of the viscosity between PM and other molasses might be attributed to their chemical composition.

Microbial qualities of PM

Microbiological analysis showed that, the total viable counts (TVC), total coliform counts (TCC) and total mould and yeast (TMY) were absent in studied PM samples (below limit of detection), which could be due to a low acidic pH that did not favor the microbial growth. The reduced microbial counts monitored in PM, could be due to the juices characteristics which have undergone a pasteurization process. The microbial load of PM is within the limits advocated by the legislation, which is 10^4 cfu/g (colony-forming units), indicating that the jams obtained were in accordance with the hygienic standards [51]. Therefore, the PM presented satisfactory health condition, meeting the sanitary standards [42].

Regarding microbiological analyzes in plum jam during storage, the results showed that no microbial growth in jams during storage time up to 12 months. Jam must be free from microbial contamination, for at least 6 months storage time [52]. The right handling may be the key to prevent microbial growth in fruit product.

The acidic pH values obtained in this study is characteristic of most fruits and this influences the shelf-life quality by restricting the micro flora to acid – tolerant microorganisms [53]. Since bacterial growth is influenced by pH of a medium, it is logical to suggest that PM could be stored longer as low pH can inhibit bacterial growth. Tounsi et al. [9] reported that the carob molasses exhibited interesting antimicrobial potential towards all the tested microorganisms. A previous study has attributed antibacterial activities of different citrus juice concentrated fruit juice to the presence of phytochemical compounds [54].

The pH in the present study is classified as acidic (pH value between 3.16 and 4.01). It has been reported that fruits with pH value of below 4.5 are a desirable trait because it halts proliferation of microorganisms in the final product [34]. Low pH in the acidic range may be an indication of good shelf life [55]. Since bacterial growth is influenced by pH of a medium, it is logical to suggest that fruit and its products could be stored longer as low pH can inhibit bacterial growth [56]. The pH reduction causes a depression of internal pH of the microbial cell by ionization of undissociated acid molecules, causing a disorder in microorganism by altering cell membrane permeability [57].

Conclusion

Traditional plum molasses, known as 'Dibss alkhookh' in Syria, was shown in this study to be a nutritional food and food ingredient. Indeed, commercial samples serve as a promising source of sugars, minerals and bio-chemicals compounds with health promoting effects, which justify their utilization in food as a natural ingredient. Syrian PM could be also exploited in food industry and used as natural ingredient or additive.

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Conflict of Interest

The authors report no conflicts of interest. The author alone is responsible for the content and writing of the manuscript.

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