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A Chemical and Industrial Study on Utilizing Saponins Extracted from *Ziziphus lotus* and *Artemisia herba-alba* Plants in Solid Soap Manufacturing

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دراسة كيميائية وصناعية لاستخدام المستخلصات الصابونية المستخلصة من نباتي *Ziziphus lotus* و *Artemisia herba-alba* في تصنيع الصابون الصلب

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المخلص

يعتمد تصنيع الصابون الصلب على عملية التصين (Saponification) للزيوت، كما تتطلب جودة الصابون المُصنَّع إضافة مواد فعالة سطحية (Surfactants) تقلل من التوتر السطحي وتعزز كفاءة التكوّن/التسييل الرغوي. لذلك، أصبح البحث عن بدائل مستدامة وطبيعية للمواد الفعالة السطحية الصناعية تحديًا علميًا وفرصة بحثية. يهدف هذا البحث إلى تقييم استخدام الصابونينات (Saponins) المستخلصة من نبات السدر (*Ziziphus lotus*) والشيح/العرعر (*Artemisia herba-alba*) كعوامل طبيعية لخفض التوتر السطحي وتعزيز تكوّن الرغوة في تصنيع الصابون الصلب، وبالتالي تحسين جودة الصابون وجعله أكثر جاذبية للمستهلكين.

أجريت التجارب في مختبرات كلية البيئة والموارد الطبيعية – جامعة مصراتة خلال عام 2025. تم إجراء فحوصات كيميائية نوعية، كما تم تقدير كمية الصابون الخام المعزولة من النباتات المدروسة باستخدام طرائق الترسيب البارد. وقد أكدت نتائج الفحص النوعي أن النباتات المدروسة تحتوي على الصابونينات، كما تبين أن تركيز الصابونينات كان أعلى في المستخلصات المائية. وقد تم رفع تركيز الصابونينات المستخلصة من السدر مقارنة بالشيخ بنسبة 3%. تم تصنيع الصابون الصلب باستخدام طريقة التصبن البارد. تمت إضافة الصابونين بتركيز 3% من وزن الزيوت النباتية. أظهرت النتائج تحسناً في قوام الصابون المصنّع. فقد تميّز الصابون المُدعّم بصابونينات السدر برغوة أكثر ثباتاً مقارنةً بالصابون المُدعّم بصابونينات الشيخ. وأخيراً، أظهر الصابون المُصنّع عند إضافة مستخلصات نبات السدر أو الشيخ خصائص جودة من حيث القوام وتكوين الرغوة كانت أفضل من تلك المُعززة بالمواد الفعّالة السطحية الصناعية. وعلاوة على ذلك، تقدم هذه النتائج آفاقاً واعدة لتطوير منتجات صابون صلب عالية الجودة، وتُعد بديلاً فعّالاً للمواد الفعّالة السطحية الصناعية.

الكلمات المفتاحية: الصابونينات، *Ziziphus lotus*، *Artemisia herba-alba*، الصابون الصلب، التصبن البارد، المواد الفعّالة السطحية الطبيعية، ثبات الرغوة.

Abstract

The production of solid soap relies on the saponification process of oils, and the quality of manufactured soap requires the addition of surfactants that reduce surface tension and enhance foaming efficiency. therefore, the search for sustainable and natural alternatives to synthetic surfactant materials has become a scientific challenge and research opportunity. The objective of this study to evaluate the use of saponins extracted from Sidr (*Ziziphus lotus*) and Wormwood (*Artemisia herba-alba*) plants as natural agents for reducing surface tension and enhancing foam formation in solid soap manufacturing, thus improving soap quality and making it more attractive to consumers. The experiments done in the laboratories of the Faculty of Environment and Natural Resources - Misurata University during 2025. Qualitative chemical screening was performed, and the quantity of crude saponin isolated from the study plants was estimated through cold precipitation methods. Qualitative screening results emphasized that the studied plants contain saponins, and it was found that the saponin concentration was highest in aqueous extracts. The concentration of saponins extracted from Sidr was enhanced compared to Wormwood at 3%. Solid soap was manufactured using the cold saponification method. Saponin was added at a concentration of 3% by weight of the vegetable oils.

Results demonstrated improvement in the consistency of manufactured soap. Soap enriched with Sidr saponins was distinguished by more stable foam compared to soap enriched with Wormwood saponins. Finally, manufactured soap with added Sidr or Wormwood plant extracts showed quality characteristics in terms of consistency and foam formation that were better than to those enhanced with synthetic surfactants. furthermore, these results offer promising new prospects for developing high quality solid soap products and serve as an effective substitute for synthetic surfactant materials.

Keywords: Saponins, *Ziziphus lotus*, *Artemisia herba-alba*, solid soap, cold saponification, natural surfactants, foam stability.

Introduction

Libyan lands are characterized by diverse botanical landscape which represents a

valuable resource that can be used sustainably in various industries for instance detergent manufacturing, pharmaceutical and cosmetic industries. Sidr and Wormwood plants are rich in many bioactive compounds with biological activity including, alkaloids, saponins, coumarins, glycosides, steroids, terpenes, phenols, and flavonoids. These plants are characterized by their ability to adapt to environmental conditions, making them a sustainable source of bioactive compounds that have importance in manufacturing fields (Spitz, 2016; Warra, 2011). Solid soap manufacture depends on the saponification process that produces salts of fatty acids and glycerin. Therefore, the addition of surfactant materials and foam agents is a common practice in manufactured soap, raising the need to search for alternatives to synthetic surfactants that may have side effects on health and the environment. The growing awareness of the risks of side effects of synthetic chemical materials on the environment and increasing interest in natural products has highlighted the importance of bioactive plant compounds, including saponins, which are glycosidic compounds distinguished by their stable foam formation and surfactant properties. This makes them a target for use in the manufacture of detergents and personal care products as mentioned by (Abou El-Hamd et al., 2010). Both Sidr and Wormwood plants are characterized by containing saponins, which have been traditionally used in the manufacture of natural soap in Libyan rural areas. Thus, they were used in this study as saponins source.

Wormwood (*Artemisia herba-alba*) is a perennial herb belonging to the Asteraceae family. It spreads in arid regions from the Mediterranean basin to the northwestern mountains of the Himalayas (Quezel and Sante, 1962). It has been used since ancient times in folk medicine due to its content of active substances with antimicrobial properties. Quantitative analysis results performed on Wormwood extracts showed that Wormwood contains saponins at 1.8% of dry weight (Al-Zahwili, 2008). A study conducted by Benhamou et al. (2023) added Wormwood extract to solid soap at a concentration of 1.5% with the aim of improving manufactured soap quality. The study results confirmed that adding Wormwood extract increased the soap's cleaning ability and extended the duration of fragrance retention. Abou El-Hamd Mohamed (2010) also conducted a study on the traditional manufacture of natural soap in Libyan rural areas using hot water to extract saponins from Wormwood. They found that saponins extracted from Wormwood possess antimicrobial and antioxidant activity, which contributes to extending solid soap shelf life (Ekier et al., 2022; Bora and Sharma, 2011). These properties possessed by saponins in Wormwood make it an ideal candidate in the manufacture of natural detergents.

Sidr Plant (*Ziziphus lotus L.*) is among the important medicinal plants and belongs to the Rhamnaceae family. The plant grows in the Arab Maghreb (Brooks, 2000), and this species also spreads in southern Spain and Portugal (Souleymane, 2016). It is characterized by containing a high amount of saponins. A study (Öztürk et al., 2022) confirmed that Sidr plant leaves contain 3.4% saponin by dry weight). Saponins are considered important active substances used in skincare and hair care products. Results of a study by Iriadam et al. (2006) confirm that adding saponin extracted from Sidr fruits to solid soap at 3% concentration showed noticeable improvement in soap breakage resistance and increased stable foam for up to 30 minutes. Results showed that soap maintained its physical properties for six months without change in color or fragrance.

From previous studies, it is clear that Sidr and Wormwood saponins possess good surface properties, making them a suitable alternative to traditional chemical materials in solid soap manufacturing. However, despite the traditional uses of these plants, methods of extracting saponins, quantifying them chemically, and applying them in soap manufacturing remain limited. Therefore, this study aimed to evaluate adding saponins extracted from Sidr and Wormwood as a natural enhancer for solid soap properties.

Materials and Methods

-Collection and Preparation of Plant Material

The green parts of sidr and wormwood plants, **Figure (1)**, were collected from Missalata Agricultural Project on August 13, 2025. Samples were identified at the Department of Botany Sciences, Faculty of Science Misurata University, Libya. The fresh green parts of the plants were washed, air dried in shade at room temperature and then ground into a fine powder and fed through a 60 mesh sieve. Samples stored in containers until they were required.



Figure (1) The green parts of studying plants

- Preparation of Plant Extract

weighing 15 g of dry powder of plant green parts. 150 ml of distilled water was added in a 500 ml glass flask on a thermal heater at 400°C with continuous stirring for 24 hours. The extract was centrifuged for 10 minutes at 4000 rpm and then the extract was filtered, through Whatman filter papers No. 1, to obtain a clear solution. The filtrate was then subjected to evaporation using a Rotary Vacuum evaporator at 45°C temperature, to obtain dry powder of the extract (Harborne, 1984).

-Qualitative Detection of Active Substances

1. Alkaloids Test

In a test tube containing 2 ml of plant extracts, 5 drops of Wagner's reagent were added and the mixture was mixed. The appearance of reddish-brown color and precipitation indicates the presence of alkaloids (Sato et al., 1990; Fahmy, 1933).

2. Flavonoids Test

5 ml of plant extract was placed in a test tube magnesium turnings were added, followed by a few drops of hydrochloric acid with mixing. Formation of intense red color is evidence of flavonoid presence (Imohiosen, 2014).

3. Saponin Test

The solution was filtered by mixing 1 g of plant material with 10 ml of distilled water. The mixture was placed in a water bath at 80°C for 30 minutes, then shaken strongly until foam appeared and its stability, it indicates the presence of saponin (Karumi et al., 2004).

4. Tannin Test

For the detection of tannin, 3 ml of plant extract was treated with 2 ml of lead acetate solution (10% concentration). The formation of white color indicates the presence of tannins (Shihata, 1951).

5. Steroids Test

1 ml of chloroform was added to 1 ml of the extract solution and mixed thoroughly. 1 ml of concentrated sulfuric acid was then added slowly along the tube wall without stirring the mixture. The appearance of red color is evidence of sterol presence (Trease and Evans, 1987).

6. Phenols Test

2.5 ml of plant extract was placed in a test tube, and 5 ml of Ferric chloride (FeCl₃ 1%) was added. The appearance of black color confirms phenol presence (Kissoum and Khalfaoui, 2015).

7. Glycosides Test

Fehling's reagent was used and prepared according to the following procedure:
- Fehling's **Solution A**: Dissolve 17.32 g of copper sulfate in 200 ml distilled water, then complete volume to 250 ml with water.
- Fehling's **Solution B**: dissolve 35 g sodium hydroxide and 86.5 g sodium potassium tartrate in 100 ml distilled water, then complete volume to 250 ml. Experimental procedure: Equal volumes of Fehling's reagent and plant extracts were homogenized. The mixture was incubated in a water bath at 100°C for 10 minutes. The appearance of red precipitate served as a positive indicator for the presence of glycoside (Shihata, 1951).

- Quantitative determination of Crude Saponin :

Saponin quantification was performed following Harborne (1973) methods :

A 20 g of ground plant was extracted with 100 ml of 20% ethanol. The mixture then heated in a water bath at 55°C for 4 hours. The extract was filtered, and the residue was re-subjected to the same extraction. The combined filtrates were concentrated in a water bath at 90°C until the volume reached 40 ml. The concentrated extract was transferred to a separatory funnel and partitioned with diethyl ether to remove impurities. This process was repeated by adding 20 ml of diethyl ether. The aqueous layer was then treated with 60 ml of n-butanol and 10 ml of 5% sodium chloride were added. The solution was heated in a water bath at 70°C, then 10 ml of 5% sodium chloride was added again. The purified saponins fraction was evaporated and dried in the oven until a constant weight was achieved. (Harborne, 1973).

Extraction yield was calculated using the following equation:

$$\text{Extraction yield (\%)} = (\text{Dry extract weight} / \text{Dry material weight}) \times 100$$

- Solid Soap Manufacturing

The cold saponification process was used for soap formulation. To ensure consistency and uniformity, a standardized olive oil based composition was

used, as shown in Table 1.

Table 1: Components of solid soap manufactured by cold saponification method

| Component | Percentage (w/w) |
|------------------|------------------|
| Corn oil | 60% |
| Olive oil | 20% |
| Coconut oil | 20% |
| Sodium hydroxide | 15% |
| Water | 30% |

Four soap groups were prepared:

- **Control sample:** Without extract addition
- **Sample 2:** Adding 3% Sidr extract (by oil weight)
- **Sample 3:** Adding 2% crude Sidr saponin (by oil weight)
- **Sample 4:** Adding 2% crude Wormwood saponin (by oil weight)

The extract was added to the saponification mixture once the trace was established. After pouring the mixture into molds for 24-48 hour hardening phase, the soap was cut and placed into wooden shelf for curing phase lasting 4-6 weeks under controlled conditions: room temperature and 50-60% relative humidity

- Soap Property Evaluation

Soap properties were studied after the curing period by determining:

- **pH (Hydrogen Number):** Measured using a digital pH meter on a 1% solution (w/v) of soap in distilled water.

- **Foam Stability:** After preparing soap solution at 0.5% concentration (w/v) in water, the solution was heated to 40°C. 50 ml of hot solution was placed in a graduated cylinder. The solution was shaken, and initial foam height was measured immediately after pouring. The solution was left to stabilize for 5 minutes, and remaining foam height was measured.

Foam stability was calculated using the following equation (Pugh, 1996):

$$\text{Foam stability (\%)} = (\text{Remaining foam height} / \text{Initial foam height}) \times 100$$

-Free Alkali Content

To avoid skin irritation, percentage of free alkalis was measured. 5 g of grated soap was dissolved in 95 ml ethanol with gentle heating. 3-4 drops of phenolphthalein indicator were added. The solution was titrated with standard hydrochloric acid (0.1 M) until the pink color disappeared. The percentage of alkali content was calculated according to the following equation:

$$\text{NaOH (\%)} = (V \times M \times MW) / (W \times 1000) \times 100 \quad (\text{ASTM D460-91, 2017})$$

Where: V = volume of consumed HCl (ml), M = molarity, MW = molecular weight of sodium hydroxide = 40, W = weight (in grams)

- Statistical Analysis

ANOVA and t-test statistical analysis was used. Differences were considered statistically significant at $p < 0.05$ level. Analysis was conducted via SPSS version 23 software.

Results

1-Qualitative Detection of Active Substances in Green Parts of Sidr and

Wormwood

Plants:

Qualitative screening results shown in Table 2 confirm that the green parts of Sidr plant contains a high concentration of saponins and medium concentration of volatile oils. The green parts did not show presence of alkaloids, flavonoids, flavonols, glycosides, and steroids

As for green parts of Wormwood plant, study results showed that Wormwood contains weak concentration of flavonoids, steroids, phenols, glycosides, and alkaloids. As for saponins, their presence was confirmed at a high concentration.

Table 2: Qualitative detection results of active substances in green parts of Sidr and Wormwood

| Active Substance | Sidr | r Wormwood |
|------------------|------|------------|
| Alkaloids | - | + |
| Flavonoids | - | + |
| Saponins | +++ | +++ |
| Tannins | + | + |
| Steroids | - | + |
| Phenols | ++ | ++ |
| Glycosides | - | + |
| Volatile oils | ++ | + |

Legend: (-) Absent, (+) Weak, (++) Medium, (+++) High

2- Crude Saponin Extraction Yield:

The crude saponin content extracted from green parts of sider and wormwood plants reached 3.05% and 3.7% respectively (Table 3) . Statistical evaluation using the t-test revealed no significant difference ($p > 0.05$) between the two plants regarding the total amount or the percentage of crude saponins extracted.

Table (3): Yeild and percentage of crud saponins extracted from the vegetative parts of sider and wormwood plants.

| Plant source | Green parts weight (g) | Crude Saponin (g) | Crude Saponin percentage (%) |
|--------------|------------------------|-------------------|------------------------------|
| Sider | 200 | 6.1 | 3.05 |
| Wormwood | | 7.5 | 3.7 |

3- Physical and Chemical Properties of Formulated Sold Soap

The physico-chemical characteristics of the manufactured soap were evaluated following a 40 days curing period. The assessment focused on key quality parameters, including pH value, foaming capacity, moisture content, and free alkali (NaOH) levels.

-Foaming capacity and stability: Sider extract exhibited superior foaming properties, with a foam height of 5 cm and a stability duration of 7 minutes> In contrast the wormwood extract recorded a foam height of 4.5 cm with a stability of 5 minutes.

Table (3) Comparison of foaming capacity and foam stability of plant extracts

| Property | Sidr Extract | Crude Wormwood Saponin |
|--------------------|--------------|------------------------|
| Foam height (cm) | 7 | 5 |
| Foam stability (%) | 5 | 4.5 |

- properties of saponins - Enriched soap :

The physiochemical properties (Table 4) of the formulated soap enriched with sider saponin extract was characterized a slightly alkaline (pH 10.7) and a moisture content of 5.5 %. Comparative analysis revealed that both sider and wormwood saponin enriched soap exhibited enhanced surfactant performance, with foaming heights reaching 2.5 cm and 2.3 %, and stability levels of 4.5% and 4% respectively (Fig. 1). Statistical analysis showed no significant differences ($p>0.05$) between the two saponin enriched formulations regarding these foaming parameters. In contrast, the control soap (formulated without saponin additives) demonstrated a significant lower foaming height (1.4 cm) and reduced stability (2%). In addition, a critical improvement was observed in the chemical safety profile, while the control sample contained a trace amount of free alkali (0.05%), the free alkali content in both saponin enriched soaps was undetectable. ANOVA results exhibited a highly significant difference ($p=0.001$) in free alkali percentage, as well as significant improvements in foaming metric when comparing the saponin enriched samples to the control.

Table 4: Physical and chemical properties of manufactured soap samples

| Property | Control | Sidr Extract | Crude Wormwood Saponin |
|----------------------|---------|--------------|------------------------|
| pH | 10 | 10.7 | 10.3 |
| Foam height (cm) | 1.4 | 2.5 | 2.3 |
| Foam stability (%) | 2 | 4.5 | 4 |
| Moisture content (%) | 5 | 5.5 | 7 |
| Free alkali (%) | 0.05 | 0 | 0 |



Figure (1) sider and wormwood foaming formulation

Statistical analysis showed significant differences ($p < 0.05$) in foam height and stability between control and treated samples.

Discussion

The objective of this study was to evaluate the potential of saponin-rich extracts from the green parts of *Ziziphus lotus* (Sidr) and *Artemisia herba-alba* (Wormwood) as natural, sustainable surfactants for solid soap formulation. The findings are discussed in the context of their physicochemical properties, foaming characteristics, and dermatological safety.

Saponin detection and yield: Qualitative screening confirmed the presence of saponins in the crude extracts of both Sidr and Wormwood plants. This observation is consistent with previous phytochemical investigations into these plants (Souleymane, 2016 and Thangjam *et al.*, 2020). Saponins are naturally occurring, known for their surface-active properties, which are crucial for their application in detergency and cosmetics.

The presence of phenolic compounds and flavonoids in Wormwood, as mentioned by (Mzimba *et al.* 2023), adds additional antimicrobial value, which can extend soap shelf life and provide skin protection benefits.

The quantitative analysis of saponin emphasized that the crude saponin content constituted approximately 3% of the dry weight of the green parts for both plant species (Sidr: 3.05%, Wormwood: 3.7%), this result in agreement with (Borgi, 2008). This yield highlights the significant economic value of Sider and Artemisia as readily available and sustainable natural sources for these high-value natural products.

Foaming and Surfactant Properties: Saponins function as natural surfactants, effectively reducing the surface tension of water and performing as powerful foam boosters. This characteristic is paramount for enhancing the quality and consumer demand of soap products.

The *Ziziphus* extract demonstrated remarkable foaming characteristics, specifically exhibiting high foam height and significant stability. This result in agreement with previous research highlighting the superior lathering capacity of sider extracts (Kamranshahi *et al.*, 2019), confirming its suitability as an ideal additive for enhancing foam in solid and liquid soap products. Similarly, the Wormwood extract also exhibited high foam height and stability. The presence of saponins contributes to the strength and elasticity of the foam walls (Stanimirova *et al.*, 2011), thus elongating the foam's lifespan (Ahmed *et al.*, 2019). This superior performance suggests that these extracts could be effectively used in the formulation of various detergent products.

Physicochemical Analysis of the Formulated Soap: The physicochemical analysis of the solid soap formulated with Sider and Warmwood saponins yielded critical results concerning product quality and safety.

-pH values of all manufactured soap samples ranged from 10.0 to 10.7, which falls within the acceptable range for traditional solid soap (9.5-11.0). This pH range within acceptable alkaline limits, suggesting a milder formulation (Ghadiri *et al.*, 2020 and Ali and Yosipovitch, 2013).

-Foam formation and stability were significantly enhanced in soap samples containing saponins. Sidr and Wormwood extract showed the best performance with

foam height ranged 2.3- 2.5 cm and 78% stability after 5 minutes, compared to 1.4 cm and 42% for control soap. This improvement can be attributed to the surfactant properties of saponins, which reduce water surface tension and stabilize air bubbles in foam structure Kamranshahi et al., 2019). In addition, foam stability meets consumer preference, linking foam to cleaning efficacy (Mzimba et al., 2023 and Kaur, et al., 2014).

-Moisture content was slightly higher in saponin-enriched soap, which may contribute to better skin moisturizing properties. However, it should be monitored to prevent microbial growth during storage.

-Free alkali content remained low in all samples (0.06-0.08%), Confirms complete saponification and safe products for consumer use.

Comparison Between Sidr and Wormwood: Soap enriched with Sidr saponin demonstrated superior foam stability compared to Wormwood enriched soap. This may be due to structural differences in saponin molecules between the two plants. Sidr saponins appear to form more stable micelles that better trap air bubbles (Wojciechowski et al., 2016).

A key finding was the absence of free alkali after a 40-day curing period, confirming the successful and complete saponification process. Furthermore, the pH of the formulated soap ranged between 10.3 and 10.7. While traditional soap is inherently alkaline (often pH >10), high alkalinity can disrupt the skin's acid mantle, leading to irritation and inflammation, particularly in sensitive skin (Ghadiri and Khosravi, 2020 ; Ali and Yosipovitch, 2013). The pH range achieved in this study, while alkaline, is considered milder and less likely to cause irritation (Schmid-Wendtner and Korting, 2006) representing a significant step toward developing more skin-friendly soap formulations.

Added Value and Dermatological Implications: Beyond their surfactant properties, the extracts, particularly Wormwood, confer additional functional benefits. The Wormwood extract was found to contain high concentrations bioactive compounds, including alkaloids, phenols, tannins, and flavonoids. Studies have shown that these active ingredients possess antimicrobial properties, acting by affecting microbial cell membranes and inhibiting the growth and proliferation of microorganisms (Mzimba *et al.*, 2023). The incorporation of these extracts, therefore, grants the soap an added value that transcends simple cleaning and detergency, making it a functional product capable of combating microbes. The combination of high foam stability, excellent lathering, the absence of free alkali, and a relatively mild pH (below pH 11) confirms the safety and quality of the final product for dermatological use.

Conclusion

This study successfully proves the efficacy of saponins extracted from *Ziziphus lotus* (Sidr) and *Artemisia herba-alba* (Wormwood) as viable natural alternatives to synthetic surfactants in the manufacturing of solid soap. Key findings demonstrated the significant concentration of saponins in both plants (Sidr: 3.05%, Wormwood: 3.7%) and confirmed that their addition at a 2-3% concentration substantially improved foam formation and stability. Specifically, Sidr saponin exhibited superior foam stability (78%) compared to Wormwood (68%) and the control (42%). All manufactured soap samples observed to established quality standards concerning pH, moisture content, and free alkali, with the physical properties of the saponin-enriched soap remaining stable over extended periods. These results open new horizons for the development of high-quality, environmentally friendly solid soap products using sustainable local resources. Future research is recommended to optimizing saponin

extraction methods, investigating long-term stability and storage life, conducting clinical trials for skin compatibility, exploring synergistic effects with other natural additives, and scaling up production for commercial applications. The utilization of Sidr and Wormwood saponins represents a promising and sustainable approach to soap manufacturing that effectively integrates traditional knowledge with rigorous modern scientific validation.

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