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Extraction of Caffeine from Spent Coffee Ground by Solid-liquid Extraction

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Abstract

The current research aims to utilize the spent coffee ground as a feedstock which is a waste material with a negative effect on the environment to extract natural **bioactive** valuable caffeine and determine the effective parameters of the extraction process efficiency in terms of caffeine concentration. The key studied parameters included extraction time 0- 150 min, temperature 25-55˚C, mixing speed (180-450 rpm), pH of suspension (4-9), and solvent type. The results of the experimental work showed that changing the pH of the suspension has a significant impact on the recovery rate of caffeine. When only water was used as a solvent, the concentration of caffeine increased from 135.061 (mg/L) to 2478.179 (mg/L) by increasing the pH of the suspension to 9. Another promising finding is that by changing the solvent type to an aqueous organic solvent, where the recovery of caffeine augmented remarkably. When 20% ethanol-water was utilized as a solvent and at the pH of 6 (the original pH) of the suspension, the concentration of obtained caffeine increased from 135 mg/L to 213 mg/L. Furthermore, increasing the ethanol percentage to 80%, rising the caffeine concentration to 464 mg/L at the same pH. adjusting the pH of the suspension to 7, resulted in rising the obtained caffeine concentration to 2386.13 mg/l with a solvent concentration of 80% ethanol.

Keywords: caffeine recovery, caffeine resources, extraction parameters, solid-liquid extraction, spent coffee grounds.

Introduction

Coffee is one of the most popular beverages worldwide. The high consumption rate of coffee required a high production rate. A total of 169.6 million 60-kilogram bags of coffee green beans were produced worldwide in the year 2020/2021, according to the International Coffee Organization, which represents a 0.3% increase over the produced amount in 2019/2020. The path to producing coffee is long and involves a number of stages which leads to the generation of many by-products such as pulp, husks, silverskin, and spent coffee grounds SCG [1]. Spent coffee grounds represent the most available and valuable by-product, where an amount of 650 kg of spent coffee grounds is generated from 1000 kg of green coffee beans turned into coffee beverages. It is produced by soluble coffee industries, domestic coffee consumption, restaurants and cafes coffee consumption [2].

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Although spent coffee grounds contain organic compounds, which makes them valuable, these compounds harm the environment, where they can be a source of greenhouse gases when decomposing and maybe change the acidity of the landfill and surrounding soil which can contribute to increasing soil pollution which is an important environmental concern [3-5]Caffeine (1, 3, 7-trimethyl-3, 7-dihydro-1H-purin-2, 6-dion) is the most substantial component that can be obtained mainly from coffee leaves and beans[6]. Also, it can be found in many plants, among other organic health-effective materials [7-9], such as tea leaves and cocoa beans [10]. It's a bioactive material and has high pharmaceutical importance due to its role in benefiting human health, where the regular consumption of coffee may reduce the risk of gallstones by stimulating the gallbladder [11]. Caffeine could also reduce the risks of developing some diseases like Parkinson's by protecting the human brain cells [12,13], and it can relieve asthma attacks [14-16], which can relieve headaches[17]. Despite caffeine's health benefits, caffeine can have some adverse health effects when consumed at high rates and is highly associated with decreasing bone density which leads to osteoporosis[18]. Also, it can cause dehydration and effects sleeping quality which causes fatigue during the day specifically when consumed through energy drinks [19,20]

On the above basis, spent coffee grounds can be considered a promising feedstock to extract natural caffeine, which is a valuable pure organic material.

Many extraction techniques have been developed with time to extract caffeine, such as Soxhlet extraction [21], supercritical fluid extraction [22], microwave-assisted extraction [23], ionic liquid-assisted extraction [24], ultrasound-assisted extraction [25], and high-pressure processing [26]. Some of these methods are proven to be efficient in recovery [27], and effective in decreasing the environmental impact of the common caffeine extraction methods. However, they require a high energy supply and expensive materials [28,29]. Therefore, solid-liquid extraction is a better method in terms of cost, especially when using nontoxic solvents such as water.

Solid-liquid extraction has been recognized as an established caffeine recovery method [30]. It is a process that involves the separation of a compound by means of an immiscible solvent. The process follows the second Fick's law of mass transfer[31] which describes the rate of accumulation (or depletion) of concentration within the volume as proportional to the local curvature of the concentration gradient. The theory of this process includes a number of phenomenological steps, any of which could be a limitation for the extraction process. The solvent transfers from the bulk to the suspension matrix at this stage on the interface of solid-liquid, a mass transfer resistance will appear, then the penetration or diffusion of the liquid will happen next, where the solvent diffuses in the pores of the solid suspension, and the dissolving process starts. Consequently, the solute will transport to the solid matrix's surface and into the solvent's bulk [32].

Several studies used an organic nature solvent to dissolve caffeine and found that the caffeine extraction process can accrue when ethanol is used as a solvent, and the recovery of caffeine was up to 4mg/g of spent coffee ground under a temperature of 60 ˚C [1]. When only water as solvent under a temperature of 20 ºC with no mixing, the caffeine extracted concentration reached 427.09 mg/L. The study reported that caffeine concentration increased to 827.95 mg/L when the temperature of the suspension has risen [33].

The current research highlighted the solid-liquid extraction of caffeine from the spent coffee ground as a feedstock to extract the valuable organic compound caffeine and reduce its upcoming negative effect on the environment as an emerging pollutant. The impact of the key parameters including pH which is a parameter that affects the solubility of caffeine yet its effect is rarely investigated for the solid-liquid extraction process, so the key parameters include: extraction time, temperature, mixing speed, pH of the suspension, and solvent type, were studied to determine its effect on the extracted caffeine concentration.

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Materials and Methods

Chemicals:

In this study, pure standard caffeine was purchased from (Fluka) of (100%) purity. Laboratory-grade ethanol (99% purity, oxoid), sodium hydroxide (99.8% purity, oxoid), and hydrochloric acid (38% purity, oxoid) were also utilized in the current study.

Plant Materials:

Spent coffee grounds were obtained from a local bakery (House of Dough, Baghdad Al-Jadriya). The preparation of coffee drinks was performed by a brewing method called the percolation method using a professional machine with high pressure. The average amount of spent coffee grounds produced by the bakery was in the range of 0.5-1 kg. Samples obtained from the bakery were stored at room temperature after drying for 4 hours in a 110 ˚C drier to reduce the water content to 43.9% water w/w.

Experimental Procedure:

Samples were prepared by suspending five grams from the dry spent coffee ground in 100 ml of water. The sample of the suspension was placed in an Incubator Shaker (model: Brunswick Scientific Co. G24 Environmental) to study the effect of the chosen parameter on the extraction of caffeine. The advantage of using an incubator shaker for the extraction is to provide a stable closed system for the extraction with constant temperate and no material loss.

The effect of temperature was studied by performing the extraction under the temperature range of 25-55 ºC. The mixing speed was also studied for a range of 180-450 rpm. The original pH of the prepared suspension was 6, the effect of suspension acidity was performed in the range of (4-9) and controlled by adding HCl 1M or NaOH 1M to obtain the required pH value. Finally, ethanol was added in different ratios to study the effect of changing the nature of the solvent from fully aqueous to organic aqueous solvent. The ratio of ethanol ranged from 20% to 80%. The time of all experiments was 1 hour, and samples of 5 ml were taken every 15 minutes. All the samples were filtered before further analysis.

HPLC Analysis:

HPLC analysis was conducted with HPLC model 1514 (Shimadzu Corporation) Zorbax Eclipse Plus 18 C Column, pore size 5µ, internal diameter 4.6nm and length 150 mm, Reverse phase – ODS and Flow rate of 1 ml/min (constant). The column temperature was at 40 ºC and UV detector set at 275 nm. The mobile phase was water: methanol (60:40) where both are HPLC grade. The injection sample volume was 10 µ.

Results and Discussion

Effect of Time:

Fig. 1 shows the effect of extraction time on the extracted caffeine concentration. The experiments were conducted at a temperature of 30 ºC, mixing speed of 250 rpm of, and L/S of 100ml/5g. The results demonstrated that the concentration of caffeine increased by increasing extraction time. The concentration increased from 16.289 mg/L to 49.539 mg/L by increasing the extraction time from 5 min to 150 min. It can be observed that the increment rate in caffeine concentration increases after the time of 40 min. Similar behavior was observed by Vandeponseele et al. , the study stated that after 15 min the increment of caffeine concentration is approximately constant [1]. The increasing extraction time provides better contact between suspension particles and the solvent. The smaller size particles 297–430 µm offer a sufficient surface area to promote the rate of mass transfer. Consequently, under conditions of 30 ºC temperature and a moderate mixing of 250 rpm, it can be noticed that the extraction process started at the beginning of the experiment time, even the first sample which was taken after 1 min showed that caffeine extraction starts and the concentration was 14.391 mg/L. This behavior of the instant start extraction process was also indicated by Lee et al.,[34] when rolling black tea particles were used as feedstock and fast extraction of caffeine accrued.

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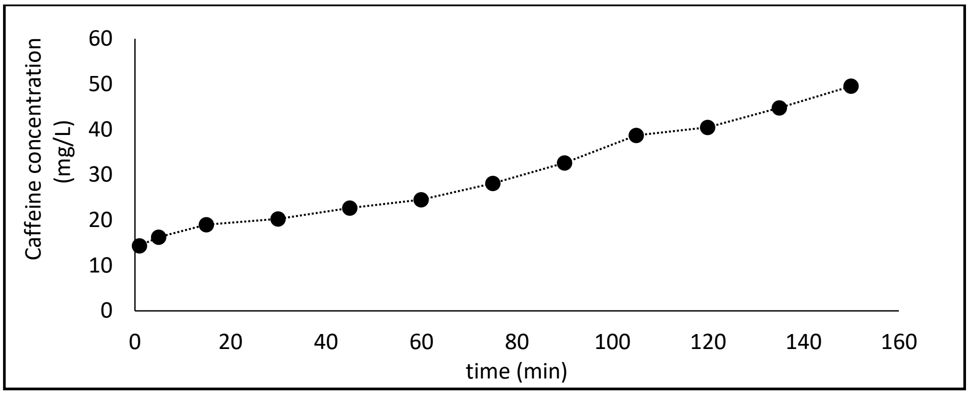

Figure 1. The effect of extraction time on extraction efficiency in terms of obtained concentration of caffeine.

Effect of Temperature:

The extraction temperature is approved to be one of the most effective parameters in the extraction process. Results in Fig. 2 show the effect of extraction on extracted caffeine concentration. The mixing speed of this set of experiments was 250 rpm, the L/S was 100ml/5g, and the results were introduced at an extraction time of 60 minutes. The results demonstrated that the concentration of caffeine in the first sample at a temperature of 25 ˚C was 24.52 mg/L, at a temperature of 55 ˚C, the concentration of caffeine increased to 132.7 mg/L for the same period of time. The current results come in line with the results obtained by Vandeponseele et al. which studied the extracted caffeine from spent coffee grounds augmented with the temperature increasing[1]. The rate of extraction increased with the rising of extraction temperature due to several factors, such as the possible rupture of the wall of the cell and the increase in the solute solubility that happens in many on heating, providing the leaching of components to the medium of the bulk according to Boateng, et al.[35]. Furthermore, higher temperatures result in reducing the solvent viscosity and enhancing the solute diffusivity by increasing the mobility of the molecules due to their increasing internal energy according to Cacace & Mazza[36].

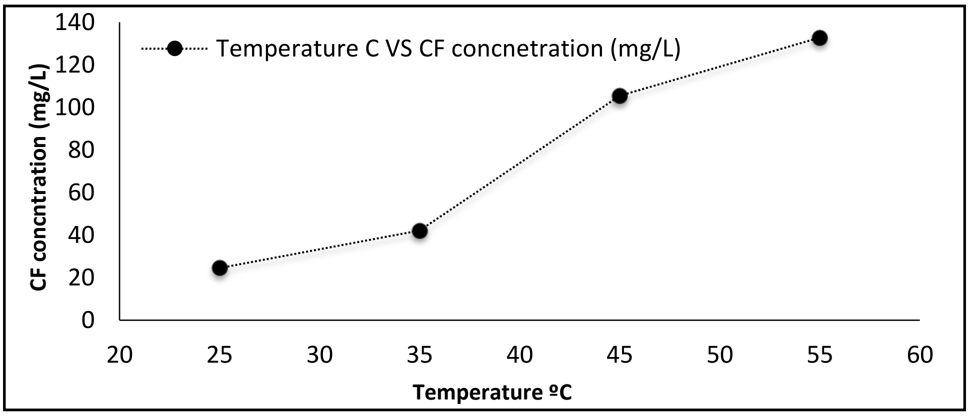

Figure 2. The effect of extraction temperature on extraction efficiency in terms of obtained concentration of caffeine.

Effect of Mixing Speed:

The mixing speed influence was studied in a range of 180, 250, 350, and 450rpm. The temperature of the process was kept at 55 ˚C, the L/S ratio was maintained at 100 ml/5g, and the results are presented at an extraction time of 60 minutes. Fig. 3 demonstrates the effect of mixing speed on extracted caffeine concentration. From the results, it can be observed that the increase in the mixing speed resulted in an increase in the concentration of caffeine. With a mixing speed of 180 rpm, the concentration of caffeine was 84.98 mg/L, by increasing the mixing to 350 rpm the concentration

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of caffeine increased to 166.96 mg/L. Furthermore, increasing the mixing speed to 450 rpm showed a decrease in caffeine concentration. This can be explained by the fact that the components are washed from the surface and later the yield is controlled by intra-particle diffusion where the mass transfer is controlled by the concentration gradient which is the driving force of the process. Therefore, when faster mixing occurs, the equilibrium will be achieved faster so less caffeine will be extracted [37].

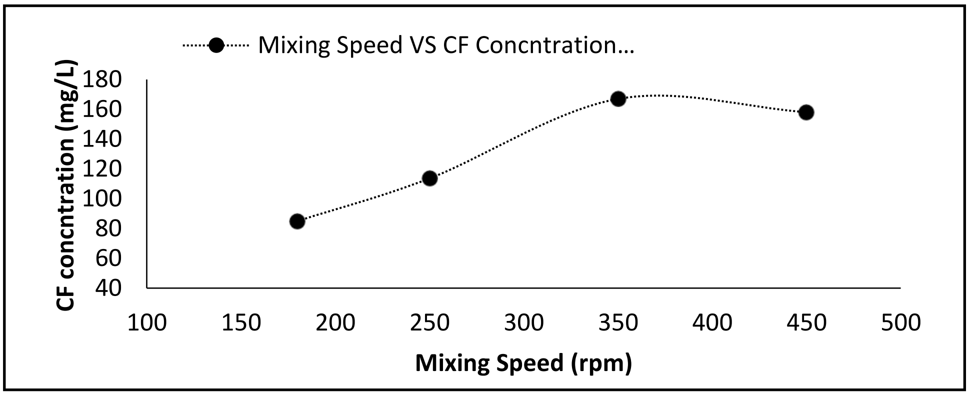

Figure 3. Effect of extraction mixing speed on extraction efficiency in terms of obtained concentration of caffeine.

liquid to Solid Ratio:

The effect of liquid to solid ratio (L/S) is shown in Fig. 4. To examine the impact of L/S on extraction performance, the temperature was kept at 55ºC and the mixing speed 350 rpm. The increase of L/S resulted in the decline of the concentration of the dissolved caffeine as shown in the figure below. When the L/S was 50ml/5g, the caffeine concentration was 503.573 mg/L, and when the L/S increased to 200ml/5g the concentration of dissolved caffeine decreased to 89.231 mg/L. the concentration of caffeine decreased with increasing the solvent volume due to the dilution that occurred from adding more of the polar solvent, the decrease in the concentration of caffeine can be explained by the nature difference between the water which is a polar compound, and caffeine which is an organic in nature compound where the process of solid-liquid extraction is a mass transfer process that is controlled by the concentration gradient and increasing the solvent volume should increase the extraction rate, however increasing the polar solvent will contribute to increasing the polarity which will result in a decrease in the solubility, this behavior can be observed when using an aqueous organic solution with an organic solvent that is less polar than water (ethanol) which increased the recovery rate of caffeine however when using methanol as the organic addition to water caffeine recovery decreased when increasing the volume of the added organic solvent this can be due to the solubility and solvent polarity with the organic nature of caffeine. the aqueous organic mixture of water/methanol cannot adjust the polarity effectively because the polarities of water and methanol are similar [38].

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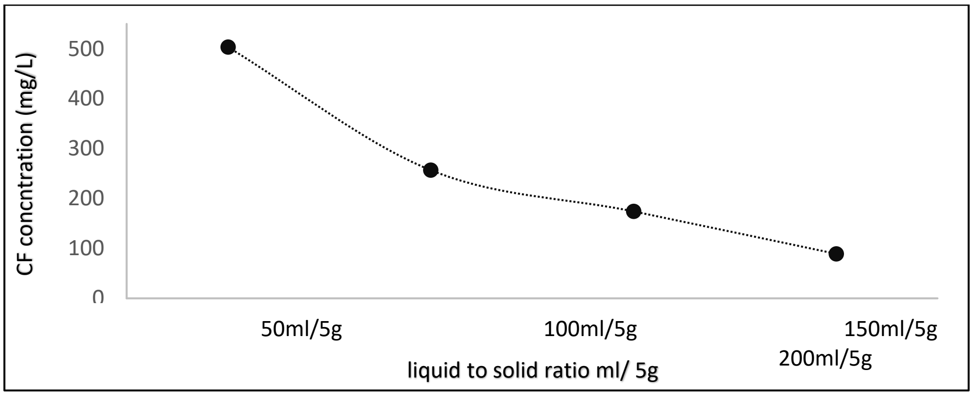

Figure 4. Effect of liquid to solid ratio on extraction efficiency in terms of obtained the concentration of caffeine.

Effect of Suspension pH:

25 the pH influence was studied for pH values of 4, 6, 7, and 9. The temperature of the process was kept at 55 ˚C, the mixing speed was 350 rpm, and the solvent-to-solid ratio was maintained at 100 ml /5g. The results are displayed in Fig. 5 which reveals that with the increasing of pH value from 4 to 6, the concentration of caffeine increased from 28.858 mg/L to 135.061 mg/L. However, increasing the pH of the solution to around 7, the concentration increased to 2348.555 mg/L and the concentration started to level off, whereas a further increase of pH to 9 enhanced the concentration slightly to 2478.179 mg/L. The pH effect is commonly studied for liquid-liquid extraction but it has rarely been studied directly regarding caffeine extraction, thus in this study an extensive approach was intended to explore the impact of pH on the process of caffeine extraction. As a result, the change in pH has shown a noticeable effect on caffeine solid-liquid extraction. The change of solubility with pH is due to the relation between pH and Pka and the protonation level of the organic compound, where caffeine is a basic compound and has a slightly higher pKa so it will show less miscibility and solubility behavior in water unlike more acidic compounds which will readily ionize in aqueous solutions[39], the hydrophilic nature of caffeine in pH values similar to the pH chosen in our study was also observed when studying the solubility of caffeine in a basic nature aqueous solution with pH values of 7-9 [40].

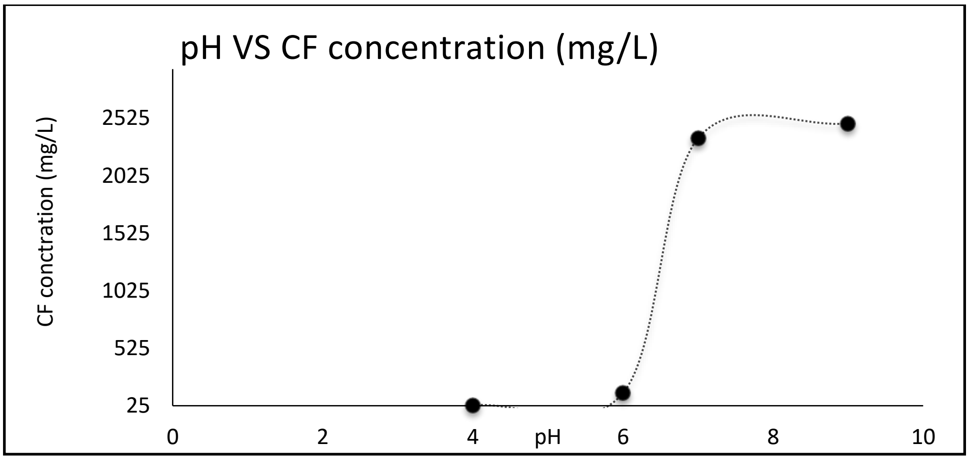

Figure 5. Effect of pH on extraction efficiency in terms of obtained the concentration of caffeine.

Solvent Type Effect

The type of solvent used for extraction has a considerable influence on the performance of extraction process. The results showed that caffeine solubility increases with the increasing of the ethanol to water ratio in the mixture as shown in the Fig. 6. For this set of experiments, the extraction time was 60 minutes, mixing speed was 350 rpm and the temperature was maintained at 55 ºC. The pH of the suspension was kept a 6. The results showed that the concentration of caffeine was 213.091 mg\L when the ratio of ethanol to water was 20:80 ml, and when the ratio was changed to 80:20 ml the caffeine concentration significantly increased to 464.544 mg\L. This increment in the extraction rate can be explained by the simple principle of "like dissolve like ", where caffeine is an organic solubility increased with the increasing of the concentration of another organic material compound in the mixture, ethanol in this case.

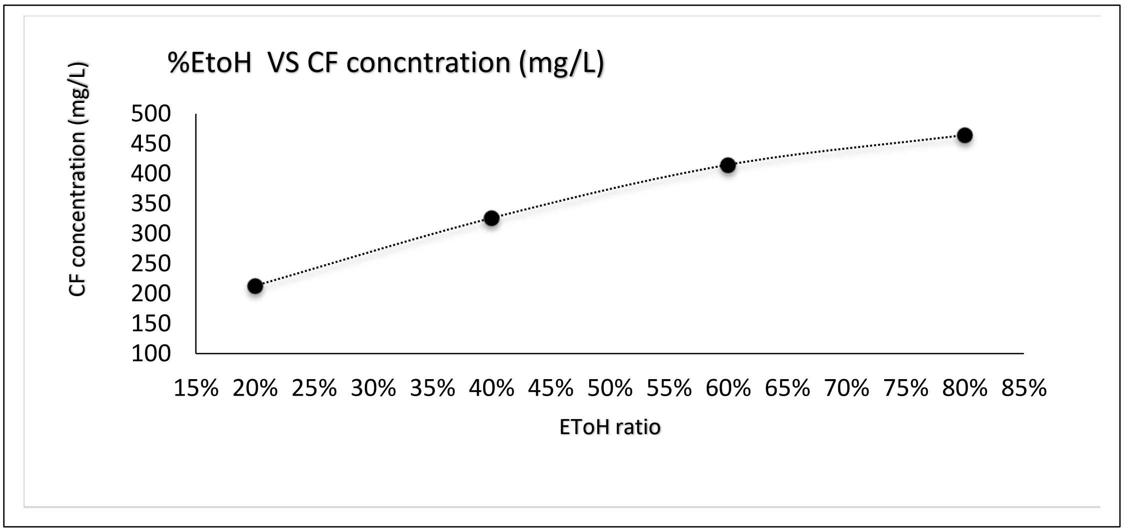

Figure 6. the effect of extraction solvent type on extraction efficiency in terms of obtained concentration of caffeine.

In addition, the interaction between the two most effective parameters (pH, solvent type) in terms of caffeine concentration was investigated to determine and compare the effect of the best extraction conditions. The extraction was performed under the condition of 55 ˚C and a mixing speed of 350 rpm and with the acidic pH =4 and a justified pH of neutral value 7, the ethanol ratio of 80% compared to the ratio of 20% under the same temperature and mixing speed. The results presented in Fig. 7 provide evidence that the ethanol ratio has an essential impact on the extraction rate regardless of the pH of the sample where the extracted caffeine concentration increased from 213.09 mg/L at (pH=6, ethanol concentration =20%) to 1829.7 mg/L at (pH=7, ethanol concentration of 20%). Symmetrically, the concentration of caffeine augmented significantly from 464.54 mg/L at (pH=6, ethanol concentration of 80%) to its maximum value of 2368.13(pH=7, ethanol concentration of 80%). This results maybe contributed to the polar nature of ethanol combined with the polar nature of water, the polarity of caffeine impact with the addition of NaOH the solution which increased its polarity and hydrophobicity [40].

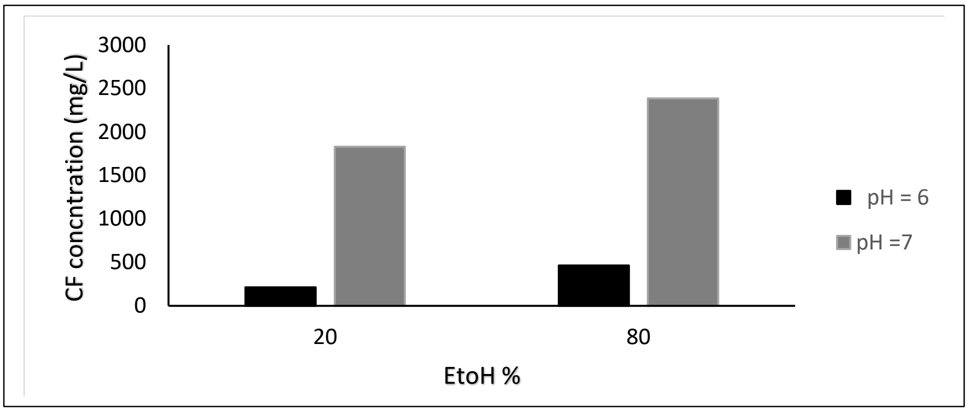

Figure 7.The effect of the best PH and solvent values on extraction efficiency in terms of obtained concentration of caffeine.

Conclusion

This research aimed to study the potential of extracting caffeine from a waste material that has a bad effect on the environment. The study focuses on the key parameters and their effect that might contribute to increasing the efficiency of the extraction process such as extraction time, temperature, mixing speed, solid-to-liquid ratio, pH, and solvent type. The results showed, that with increasing time caffeine concentration will increase where at a time of 5 min the concentration was at 16.289 mg/L and increased to 49.539 at the time of 150 min, as for the temperature effect same increment behavior was obtained by increasing the temperature from 25 °C to 55 °C which increased caffeine concentration from 24.52 to 132.7 mg/L, the mixing speed increasing also resulted in increasing extraction efficiency where extracted concentration increased from 84.99 to 166.96 mg/L when it was increased from 180 to 350 rpm, solid to liquid ratio effect showed the effect of the water polarity on decreasing the solubility of caffeine where increasing the ratio from 5g:50 ml distilled water to 5g:200ml distilled water resulted in reducing caffeine concentration from 503.573 to 89.231 mg/L, the effect of the pH displayed that caffeine solubility increasing when the nature of the solution was approaching a basic nature pH where increasing pH value from 4 to 9 increased caffeine concentration from 28.858 to 2478.179 mg/L, the parameter of solvent type results showed that increasing EToH ratio from 20% to 80 % increased extraction concentration from 213.091to 464.544 mg/L. Furthermore, the combined effect of the addition of ethanol with altering the pH of suspension shows that with a pH value of 7 and an ethanol ratio of 80% the caffeine, recovery reaches up to 2368.13 mg/L which is noticeably higher than the caffeine recovery under the same ethanol ratio of 80% with a pH of 6 of the actual suspension which was 464.544 mg/L and this highlights the significant effect of pH on the extraction process.

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Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.

- Ethical Clearance: The project was approved by the local ethical committee at University of Baghdad.

Authors' Contribution Statement

A.M. designed the study and S.R. performed all the experimental work under the supervision of A.M., both writers contributed to the analysis of the results and to the writing of the manuscript.

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استخلاص الكافيين من مخلفات القهوة بعملية الاستخلاص الصلب-سائل

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الخلاصة

يهدف البحث الحالي إلى الاستفادة من القهوة المستهلكة , كمادة وسيطة حيث تعد القهوة المستهلكة من المخلفات المضرة للبيئة الاستخراج الكافيين الطبيعي والذي يعد مادة ذات نشاط حيوي واهمية, وتحديد العوامل الفعالة في كفاءة عملية الاستخلاص من حيث تركيز الكافيين. تضمنت المتغيرات الرئيسية المدروسة وقت الاستخلاص 0-150 دقيقة ، ودرجة الحرارة 25-55 درجة مئوية ، وسرعة الخلط 180-450 دورة في الدقيقة، ودرجة الحموضة العالق (4-9) ونوع المذيب. أظهرت نتائج العمل التجريبي أن تغيير درجة حموضة المعلق له تأثير كبير على معدل استرجاع الكافيين. عند استخدام الماء فقط كمذيب ، زاد تركيز الكافيين من 135.061 مجم / لتر إلى 2478.179 مجم / لتر عندما تم زيادة الاس الهيدروجيني للعالقِ إلى9. وهناك نتيجة واعدة أخرى وهي أنه من خلال تغيير نوع المذيب. إلى مذيب عضوي مائي ، زاد استرداد الكافيين بشكل ملحوظ. عندما تم استخدام 20 ٪ من الإيثانول ماء كمذيب وعند درجة الحموضة الأصلية للمعلق ، زاد تركيز الكافيين الناتج الى 213 مجم / لتر . علاوة على ذلك ، زيادة نسبة الإيثانول إلى 80٪ ، رفعت تركيز الكافيين إلى 464 (ملجم / لتر). أدت زيادة الاس الهيدروجيني للعالق إلى7 لزيادة تركيز الكافيين الناتج إلى 2386.13 ملجم / لتر بتركيز مذيب بنسبة 80٪ إيثانول.

الكلمات المفتاحية: استرجاع الكافيين ،مصادر الكافيين ، متغيرات الاستخلاص ، الاستخلاص السائل-الصلب ، بقايا القهوة.