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Analysis of Acrylamide Levels in Various Food Types in the Iraqi Markets Using Chromatography Techniques

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Abstract

This article show the progress and application of the SPE (solid phase extraction) and HPLC (higher pressure liquid chromatography)as analytical method for the detection of acrylamide (Acr) in variousconsumable food kindsin Iraq. The chromatographic method includes, extraction and separation of acrylamide using C_{18}as a chromatographic column, followed by quantitative determination of the extracted acrylamide using known concentration standard. This method of analysis show the values of the recoveries around 83% ± 3. A suitable sensitivity value was found with an instrumental LOD and LOQ (limit of detection and quantitation) ranged from 21 to 67 μgL^{-1}, respectively. The detection of Acr was effectively achieved using homemade and commercially supplied Iraq-food samples. The results obtained show that the Acr conc. Averaged from 160µg per kg of food to 400 µg/kg of foods. The obtained numbers were inside the allowable concentration.

1. Introduction

Acr consider to be one of the synthetic monomer which is usually applied in industrial process[1,2] typically as a pre-cursor in synthetically process of poly-Acr plastics and can be used as a filling agent (reagent) in cement, also it being used for purification of drinking water and in the treatment process of waste water[3,4]. (IARC) which is the international agency of researches that specialized in cancer researches has considered Acr as a likely carcinogenic for humans, also WHO (World-Health Organization) has generated a guideline for water quality regarding Acr, they determine the value equal to 0.5 or below mgl^{-1} in drinking water is safe concentration[5,6]. The released document by SNFA (Swedish national administration of foods) in 2002,show that the Acr majorly formed in carbohydrate food by heating, the Acr in food become an necessity human health matter. A large number of methods have been established for Acr quantitative determination in different foods. Notwithstanding the necessity for methods that have sensitivity and fastness for quantification of Acr in food, because low limits stated by lawmaking organizations, just small number of methods were established mainly for that aim[7,8]. There are 2limits recognized for Acr; the first, regardwater used for drinking[9], whereas the second includes the Acr migration from materials that used for packaging into different foods type [10]. The other is stated not obvious within a LOD (detection limit) equal to ten µg Acr per kg of food, while the daily intake of about 10 of micro gs can be expected that depends on the habits of dietary. This will raise the concerns of the food manufacturers as well as the authorities for food control such as MHE (the Ministry of human Health and Environment in Iraq).

This article emphases on the quantitative determination of Acr in food in IraqHPLC-Uv, Although the published articles report the occurrence of Acr in several foods kinds [9], afew information about the actual concentration of Acr in foods in Iraq. This data will providean attention to the customers also to the Iraqi food exporters, and thus will provide local and international effect.

2. Experimental

2.1. Material and Chemicals

All used in purification process were high pressure liquid chromatography grade and were purchased from Sigma. Double distillated water were used throughout the work. Acr was bought from Aldrich that have a CatLog number equal to A8887. The samples of foods were gotten from local or homemade sources. SPE (solid-phase extraction) cartridge was type $C_{-18}$and obtained from Agilent company.

2.2. Stock solutions preparation

100 µg mL^{-1}Acr stock solutions was made by dissolving around 1 milligram of the Acr in 10.0 milliliter of distilled water. The prepared solutions saved in no light place at 4°C. before using the solutions they monitored using ultra violet spectrophotometer to check the possibility of getting photo degradation effect. The optimum time for using the prepared solution was found to be around six months. Using serial dilution, the Acr concentration prepared daily.

2.3. Preparation of samples

around ten grams of well mixed food samples were located in 100 milliliter of plastic tube and an extraction process was done using with 100 milliliter of water by intensive shaking for around half hour using shaker, then the solution was centrifuged for twenty minutes using 9000 as round per minutes.

2.4. Clean-up process

Solid-phase extraction cartridges were previously conditioned with threemilliliter methanol then equilibrated with six milliliter of distilled water. The methanolic water and the water portions were used to get the cartridge ready to use weren't re-cycled. Around two milliliter aqueous filtrate was straight loaded on the top of the Solid-phase extraction cartridges at a flow rate of twomilliliter per minutes. The solution of the extract then allowed to move over the Solid-phase sorbent bed then 0.5 milliliter of H_2O were used. The first 0.5 milliliter coming out from the Solid-phase tube was consider as waste, whereas the next one milliliter was gathered and further cleaned up usinga twenty μm filter paper then punctually injected into High-pressure liquid chromatography system for analysis.

2.5. Uv-Vis spectroscopy

the absorption spectrumfor Acr was obtained usinga singly beamed spectro-photometer model UV1650 Japan, Shimadzu, the instrument was operational with xenon type light source with a constant value of band-pass equal to 2nanometer. All the readings obtained were generated using a quartz cuvette that has a path-length equal to 1 centimeter.

2.6. Instrumentation of theHigh-pressure liquid chromatography (HPLC)

The High-pressure liquid chromatographicsamples analysis were obtained with a system controlled via computer made by Shimadzu in Kyoto, Japan, the system contained of a SCL10AVP controller. The High-pressure liquid chromatographic system has a LC-10 AVP gradient typed pump, with SPD-10AVP ultraviolet detector, and DGU-RA which is a nonlinear degasser. The chromatographic separation was done using a chromatographic column type C_{18}that has fifteen centimeter in length, 4 millimeteras a diameter, and fivemillimeter ofparticle size. The sample inject volume were kept at twenty milliliter.

3. Discussion andResults

Researches show that the raw food itselt dosent show any amount of Acr., the compound formed when foo rich in starch are cooked using temprature more that 125 C^0, a chemical reaction will happen between some sugar and Aspa (asparagine) causeing Acr. The researches show that certain cooking methods like frying, roasting and baking are the most cause of Acr. That why several attempts have been done to create accurateexperiments for the quantification of Acr. (Figure 1) in severaltypes of food.

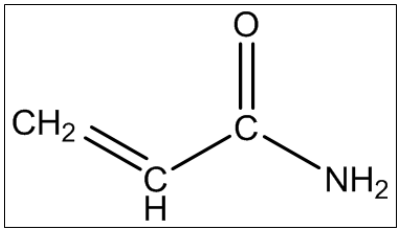

Figure 1: structure of Acr.

Several samples of foods from several brands were purchased or made in Al-diwaniyah city, Iraq, these types were, chips of potato, meat in can, canned vegetables, pasta, baked type potato and cocoa. The brands names of samples were (Hallla, Albustan and Elkaseeh). These samples were extracted and then analyzed using chromatographic method.

3.1. Ultraviolet-vis spectrum of Acr.

onemilliliter of the 100 ppm stock solution was diluted one hundred times with double-distilled water, and the Ultraviolet spectra was obtained via taking one milliliter of the sample after dilution. The wavelength scan was two hundred to four hundred nanometer using H_2Oas blank. The Acr spectrum was detected the maximum λ was 273nanometer (Figure 2).

3.2. High-pressure liquid chromatography analysis

For HPLC-Uvchromatographic separation, a commonly used type C_{18} column has been used to achieve the analysis using the same method in the previous article stated by Agilent technologies [11]. The chromatographic isolation was reached using a 100 percentacet (acetonitrile)as a moving solvent, the rate of the solvent flow was equal to onemilliliter per minute and fixed column temperature equal to 25 °C. Figure three shows a typical High-pressure liquid chromatographychromatogram of 1 ppm solution of Acr. in water, using 273 nanometer in the ultraviolet detector.

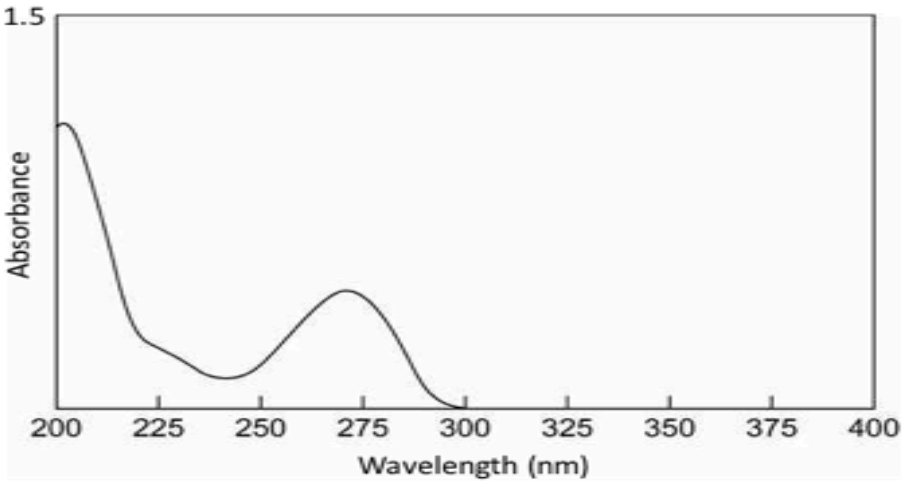

Figure 2: Ultraviolet absorption spectrum of Acr.one ppm in 100 percent acet.

Figure 3 shows a typical High-pressure liquid chromatographychromatogram of 1 part per million of solution of Acr. in water, using 273 nanometer in the ultraviolet detector. The chromatogram show a single peak and that give an indication of purity of the standard soultion used during the analysis. Also the chromatogram shows a sharp peak.

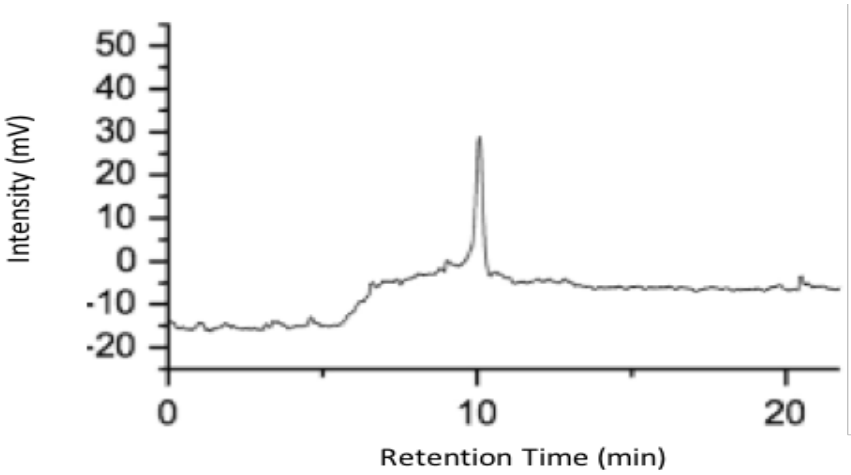

Figure 3: High-pressure liquid chromatographychromatogram obtained from 20µL injection of 1 ppmAcr.

3 chromatographic runs using the identical solution werelisted from 3 separate injections of twenty µL aliquots which provideten ± 0.5 minutes as a time of retention in the chromatogram. To build-up a calibration curve, 5 dissimilar concentrations of Acr. were made in double-distilled H_2O and then injectioninto High-pressure liquid chromatographysystem (Figure 4).

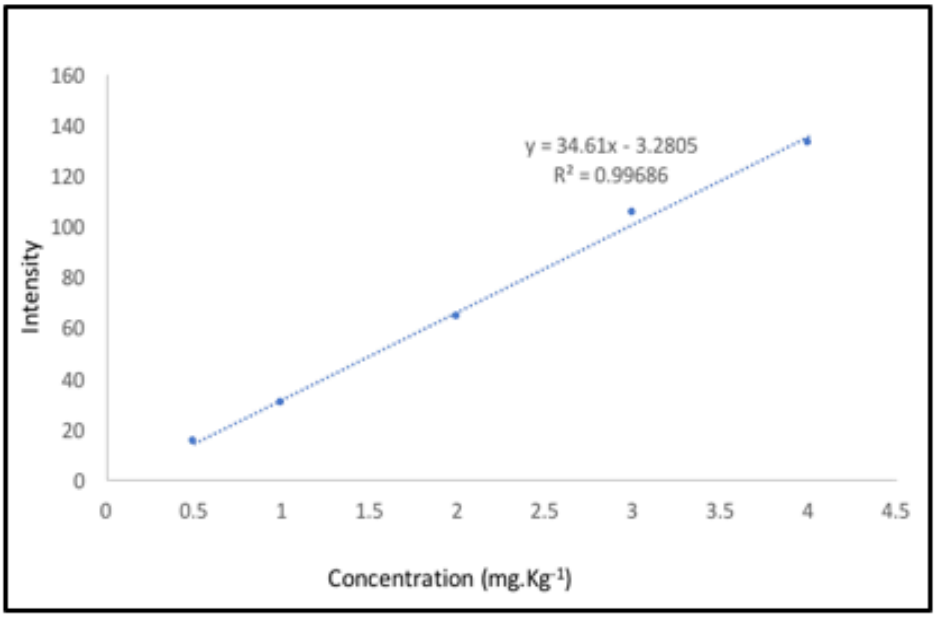

Figure 4: Calibration curve of Acr. in High-pressure liquid chromatographyusing 5 different concentrations

3.3. Quantification of Acr. in foods

Commercially obtainedand homemade samples of several foods samples were collected from the local markets, these include; chips of potato, pasta, baked type potato and cocoa products were cleaned up using solid phase extraction cartridges then HPLC determined for Acr. content. The average value for the recovery was found to be around 83 percent ± three and the LOD which is the detection and quantification limit (LOD and LOQ, where limit of quantification defined as 3.3 limit of detection.) were twenty one and sixty sevenppb respectively using sixteenvalues for the blank to estimate limit of detection.

The higher level of Acr. Was found in the Chips of the potatoaround 392 µg per kg. Acr.concentration in various products is recorded in **Table 1**.

Table 1: Acr. concentration obtained in several foods types

Foods type	**Acry. µg kg^{-1}**
Chips of potato (home made)	390 ± 13
Chips of potato (commercial)	412 ± 23
Pasta	201 ± 15
Baked type potato	301 ± 18
Cocoa (bar)	190 ± 22
Cocoa (powder)	186 ± 16
Meat in can	210 ± 21
Canned vegetables	160 ± 13

Conclusion

In this article aneffective straightforward procedure have been applied for the quantified determination of Acr. in several foods using solid phase extraction cartridge and HPLC-Uv. The results obtained show that using highe perforamnce liquid chromatography with ultavilot detector is good technque for quantification of Acr. In food while using solid pase reverse phase cartridge is such a good method of purification of Acr. prior to injection into the HPLC .The examinations and the resullts achieved in this article bring the scientists a slight closer to determine the actual content of Acr. by mimizingthe causes of the error such as the partial extraction process. Also the otained results will help the public to give more attention about chosing the optimum temprarure to cook and the way to do that.

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