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Quantitative Analysis of Components of Heroin Seized in Kuwait by Gas Chromatography/Mass Spectrometry

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Authors' contributions

This work was carried out in collaboration between all authors. All authors read and approved the final manuscript.

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Original Research Article

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ABSTRACT

Aim: To identify the main and additive components of heroin trafficked in the State of Kuwait, and to provide a satisfactory explanation for the increase in deaths among heroin addicts.

Study Design: Selected samples of non-pure powder heroin seized in Kuwait and a set of pure heroin standards have been analyzed in the State Forensic Laboratories.

Place and Duration of Study: All analyses were conducted during 2012–2014 in the Forensic Laboratories of the General Department of Criminal Evidences – Ministry of Interior – State of Kuwait.

Methodology: A total of fifty samples of non-pure powder heroin seized in Kuwait during the year 2012 and ten pure standards have been analyzed by using Gas Chromatography/Mass Spectrometry (GC/MS).

Results: The concentration % of the diacetylmorphine in Kuwait is in the range of (20-40%) and is much less than that manufactured (Her) in Afghanistan and Southeast Asia (concentration% (67-

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80%) and (85-95%) respectively. All heroin samples contain papaverine, noscapine, acetylcodaine and 6-mono-acetylmorphine with range proportion 0-40%, 2-38%, 4-40% and 4-40%, respectively. However, morphine has not been detected in all samples. The additive substances (adulterants) have been detected in heroin samples in various percentage concentrations with the following range concentrations %: caffeine % 0.54-16%, diazepam 0.02-7.3%, and in some samples: paracetamol (acetaminophen) 0-21.4%, (phenobarbital) 0-74% and (*Alprazolam*) 0-46.2% (new additive).

Conclusion: The illicit heroin trafficked in Kuwait as diacetylmorphine has concentrations ranging from 20% to 40%, and its main components include papaverine, noscapine, 6-monoacetylmorphine and acetylcodaine, while morphine is absent. In addition a number of additive substances have been recorded, including paracetamol (acetaminophen), caffeine, diazepam, phenobarbital and Alprazolam as new additive substance.

The similarity between the additives identified in heroin under study and those recorded in heroin manufactured in Afghanistan and Iran would suggest that the mixed heroin in the two countries are possible sources of heroin seized in Kuwait.

Keywords: Heroin (diacetylmorphine); forensic; additive substances; gas chromatography/mass spectrometry (GC/MS).

1. INTRODUCTION

Kuwait is considered as a consumer country for many types of drugs, which are smuggled from abroad through all outlets: air, sea and land. As a result of its central geographical location between producing countries such as Western Asia, South West Asia, Europe and some African countries, Kuwait has been a commercial route between these drugs producing countries and consumer countries. However, Kuwait is not producing, manufacturing or cultivating any narcotic substances [1].

Heroin is the most prevalent type of drugs in Kuwait as the statistics of the Forensic Laboratory of the Ministry of Interior show a considerable increase in the amount of heroin seized and the number of deaths of addicts from the drug in the year 2012 [2].

Heroin is the most important and most dangerous derivative of opium and is quickly addictive gravity, as it exceeds its ability to anesthesia ten times the capacity of a morphine component. Heroin is a highly toxic substance and a killer. Consequently, it is an illegal substance listed in the drug Schedules 1 of Kuwait [2-5].

Clinical effects can be due to heroin itself, but also due to the impurities or substances used to "cut" the drug. Many research studies on heroin overdose have shown that most of these deaths occur in the company of other people. Possibly the most important finding to emerge from heroin overdose research is the role of *polydrug* use.

The extensiveness of polydrug use among "heroin" overdoses suggests that polydrug toxicity is a better description of the toxicology of the overdose. The majority of drugs associated with an increased risk heroin overdose are alcohol, benzodiazepines, and tricyclic antidepressant. So the primary risks of chronic intoxication are physical and psychological dependence, systemic complications due to heroin, adulterants and multiple drugs abuse [6,7].

A series of detailed investigations using test colour, Thin Layer Chromatography (TLC), and Gas Chromatography/Mass Spectrometry (GC/MS) techniques have been carried out on selected number of non-pure heroin samples to study in depth the physical and chemical characteristics of heroin trafficked in the country. Based on colour it was possible to divide the samples into four groups; namely, yellow, light beige, light brown, and beige [8].

The present work throws light on the main components of heroin seized in Kuwait and the additive substances present in it.

2. EXPERIMENTAL WORK

Selected samples of non-pure heroin seized in Kuwait and pure standard samples have been analyzed in the Narcotics and Psychotropic Substance Section (NPSS) (the Forensic Laboratories, Ministry of Interior, Kuwait) by using Gas Chromatography/ Mass Spectrometry (GC/MS) in accordance with analytical methods described by the Manual for Use by National

Narcotics Laboratories and Clarke's Analysis of Drugs and Poison, for qualitative and quantifying all drug components additive to seized heroin [4, 9,10].

2.1 Sample Preparation

2.1.1 Pure standard samples

Ten external pure samples have been supplied Alltech Associates namely, from Inc.; "Diacetylmorphine(Her), Morphine (Mor), Papaverine (Papa), Noscapine (Nos), Acetylcodaine Caffeine (A.cod), (Caf), Paracetamol (Para) Diazepam (Dia), Phenobarbital (Phe), and Alprazolam (Alpra)" [11].

2.1.2 Non-pure samples

Fifty non-pure powder samples of heroin have been collected from the cases of seized heroin investigated by the Forensic Laboratories during the year 2012. The samples were divided into four groups according to their colour [8].

All pure and non-pure samples have been subjected to the same conditions: each sample is 20 mg in weight, dissolved in 2 ml of Methanol (MeoH), and filtrated to injection with 1 μL in GC/MS.

2.1.3 Apparatus used for measurements

Gas Chromatography/Mass Spectrometry (GC/MS) has been used to analyze the heroin samples and standards at the same conditions as mentioned above.

The following operating conditions have been applied in accordance with Agilent 6890 series Gas Chromatography "Operating Manual" [12]:

Operating conditions:

The following operating conditions are in accordance with Agilent 6890 Series Gas Chromatography "Operating Manual" [12]:

GC/MS Agilent Technologies 6800 N (integrated LAN) liquid samples and 5973 inert MS (mass selective) single quadurpole detector, with MSD in EI mode (electron ion source) and column (ULTRA 2) with 30 m x 0.32 mm x 0.52 μm with temperature limits from -60°C to 350°C with automatic sampler compone.

Carrier gas (He) Helium with flow rate: 1.4

Inlet = spilitless with 280°C. Ramp (temperature Column Initial: 80°C hold 1.00 min

Ramp: 10°C-280°C hold 9 minutes (for 30

minutes)

Post: 300°C hold 10 minutes Run time (40

minutes).

Pressure: 1.68 psi

Interface temperature: 270 (AUX)

Injection Volume: 1.0 µl by syringe size 10.0 µl.

3. RESULTS

GC/MS is a well-known technique commonly used in the Forensic Laboratories in Kuwait for the analysis of seized heroin samples to detect the diacetylmorphine and the additive components mixed with it.

3.1 Groups of Samples Collected

The fifty heroin samples are divided into four groups according to their colour; namely Group 1 with yellow colour includes two samples, Group 2 with light beige colour includes 14 samples, Group 3 with light brown colour includes also 14 samples and Group 4 with beige colour includes 20 samples; all samples have been compared with the results of the standards analysis and the concentration of the additive components in all seized heroin samples has been calculated.

3.2 A Detecting the Components in all Samples

From the results of GC/MS analysis of all fifty samples present in the four groups it is clearly that the main components of seized heroin in Kuwait are acetylcodaine, monoacetylmorpheine and diacetylmorphine in all fifty samples (Fig. 1). Group 1, which consists of two samples, records the presence of Caf. Dia and Nos in one sample with the same proportion 2% of total samples, while Para, Phe and Papa are not detected in this group. Consisting of fourteen samples Group 2 contains Para in three samples with proportion 6% of total samples, Caf in twelve samples with proportion 24%, Papa in thirteen samples with proportion 26% of total samples. Dia in twelve samples proportion 22% and Nos in fourteen samples with proportion 28% of total samples, while Phe and Alpra are not detected also in this group. Group 3 comprises fourteen samples and records Para in five samples with proportion 10% of total samples, Caf in thirteen samples with proportion 26%, Dia in samples with proportion 24%, Nos in twelve samples with proportion 24%, and Papa in eleven samples with proportion 22%, while Phe and Alpra are recorded for the first time in this group; Phe in two samples with proportion 4%, and Alpra in one sample with proportion 2% of total samples.

Group 4, which includes twenty samples, records the presence of Para in eight samples with proportion 16% of total samples, Caf in twenty samples with proportion 40%, Papa in all twenty samples with proportion 40% of total samples, Dia in fifteen samples with proportion 30%, and Nos in nineteen samples with proportion 38%, in addition to the identification for the second time of Phe and Alpra, as Phe was detected in four samples with proportion 8%, and Alpra was recorded in one sample with proportion 2% of total samples.

3.3 Calculating the Concentration of Diacetylmorphine

The peak area for both the standards and seized heroin samples have been used to calculate the concentration of basic substance the diacetylmorphine (Her) in all fifty samples (all groups) as shown in Fig. 2. Group 1 includes two samples with (Her) concentration (conc.) 3.139 mg/ml. Group 2 includes fourteen samples with (Her) conc. 43.435 mg/ml marking the highest concentration recorded. Groups 3 also includes fourteen samples with (Her) conc. 26.316 mg/ml which is less than that in Group 2, while the concentrations of (Her) in Group 4 which include twenty samples, is 42.836 mg/ml.

3.4 Calculating the Concentration of Additive Substances

Figs. 3 and 4 summarize the additive substances identified in the fifty seized heroin samples. They include Caffeine, Paracetamol, Diazepam, Phenobarbital and Alprazolam as adulterant components with the following concentrations: Group 1 contains Caf with concentration 0.274 mg/ml and Dia with concentration 0.01mg/ml, while para, phe and alpra are not included in this group. Group 2 includes Para with conc. 0.13 mg/ml, Caf with conc. 7.415 mg/ml and Dia with conc. 3.674 mg/ml, while Phe and Alpra are not included either. However, Group 3 includes para with concentration (10.699 mg/ml) which is much higher concentration than that in the other groups, Caf with conc. 5.604 mg/ml, and Dia with conc.(0.89 mg/ml) which is much less than Group 2, and records the first appearance of phe with conc. 0.33 mg/ml and alpra with very high concentration of (23.083 mg/ml). On the other hand Group 4 contains para with concentration "3.806 mg/ml" which is higher than that in Group 3, Caf with conc. "8.048 mg/ml" also much higher than in the other Groups (1, 2 and 3), Dia with conc. 2.229 mg/ml, and the second appearance of Phe with conc. 0.374 mg/ml, and alpra with a concentration of 3.561 mg/ml which is less than its concentration in Group 3.

4. DISCUSSION

The characteristics of the seized heroin in Kuwait depend on the purity of heroin (diacetylmorphine) and the additive substances included.

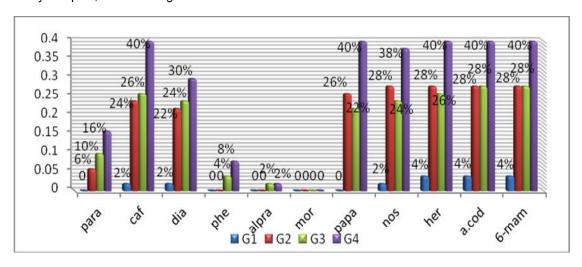


Fig. 1. The proportion of the components present in all samples (all groups)

4.1 General Characteristics of Seized Heroin in Kuwait

The heroin trafficking in Kuwait is in the form of affine powder, with different colours, ranging between different degrees of the beige colour.

As the more prevalent colour the beige colour represents the highest proportion (40% of total samples), followed by light beige colour and light brown colour with the same proportion 28% of total samples.

4.1.1 Purity of heroin (diacetylmorphine)

All the fifty samples include diacetylmorphine with different concentrations. Fig. 5 shows that the highest percentage concentration of it is in group 2 (87%) with light beige colour and in group 4 (86%) with beige colour which is the largest group (20 samples) with the dominant colors in heroin trafficked in Kuwait. This is

followed by light brown colour with conc. % 52.6%. This difference is attributed to the method used in the manufacture of heroin and to the different additives added to it. For example, heroin manufactured in Afghanistan has much higher concentration% of 80% (Her) than that seized in Kuwait "up to 40%" [13,3].

4.1.2 The basic components of seized heroin in Kuwait

The results for analysis of the fifty samples recorded in Fig. 1 show that the basic components include papaverine, noscapine and acetylcodaine as derivative of opium, and 6-monoacetylmorphine as derivative of heroin during its manufacturing with various methods. With the exception of acetylcodaine and 6-monoacetylmorphine which are present in all samples (100%), the other components (papaverine & noscapine) are recorded in the range of 0-40%, and 2-38%, respectively.

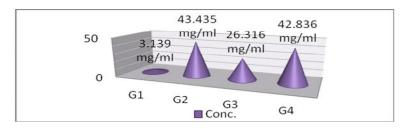


Fig. 2. The calculated total concentration (mg/ml) of heroin in all groups

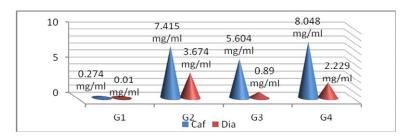


Fig. 3. The calculated total concentration (mg/ml) of the additive substances (caf & dia) in all groups

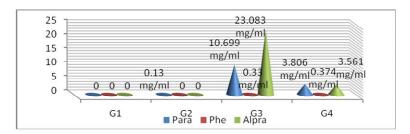


Fig. 4. The calculated total concentration (mg/ml) of the additive substances (para, phe & alpra) in all groups

4.1.3 The additive (adulterant) substance in seized heroin in Kuwait

The seized heroin in Kuwait is mixed with other substances as shown in Fig. 6; namely paracetamol, caffeine, diazepam, phenobarbital and alprazolam. These substances differ in their appearance and concentration% in all groups.

As in Fig. 7 the results show that each of the two Groups 2 & 4, which possess different degrees of beige colour, contain the highest concentration of (her) (34.44 mg/ml in Group 2 and 42.84 mg/ml in Group 4). Also included is caffeine with conc. (7.42 mg/ml in group 2 and 8.05 mg/ml in Group 4) and diazepam with concentration (3.67 mg/ml in group 2 and 2.23 mg/ml in Group 4) higher than other groups.

Fig. 8 shows that Group 3 with light brown colour contains paracetamol (10.7 mg/ml), and alprazolam (23.1 mg/ml) which are representing

the highest concentrations of paracetamol and alprazolam than the other groups.

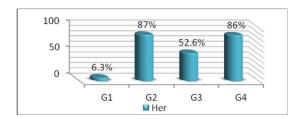


Fig. 5. The concentration% of diacetylmorphine (Her) in all groups

Comparing the additives of heroin seized in Kuwait to that of Iran which cultivates and manufactures the drug, the Iranian heroin is mixed with the drugs benzodiazepines (Diazepam, Alprazolam and Clonazepam) and barbiturates (phenobarbital) with caffeine and other opium alkaloids [14].

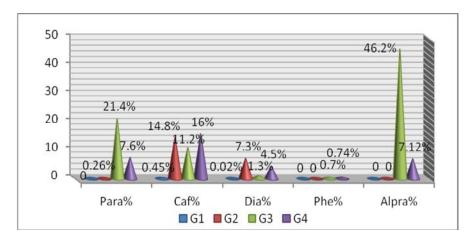


Fig. 6. The percentage concentration of all additives in all groups

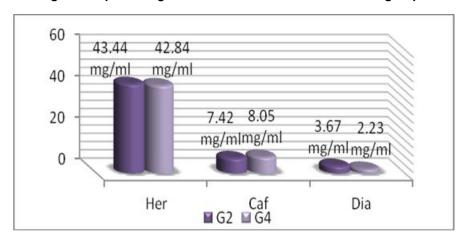


Fig. 7. The highest concentrations of additive substances in Groups 2 and 4

In Afghanistan, the heroin is also mixed with a number of psychotropics substances including diazepam and phenobarbital. This strongly suggests that the mixed heroin in Iran and Afghanistan are possible sources of heroin seized in Kuwait [14,15].

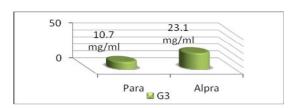


Fig. 8. The highest concentrations of additive substances in groups 3

5. CONCLUSION

A total of 50 selected non-pure powder samples of illicit heroin trafficked (seized) in the State of Kuwait, and a set of pure white powder heroin standards (free from any additives) have been analyzed by GC/MS in order to identify the main and additive components of heroin trafficked in Kuwait.

The concentration percentage of diacetylmorphine in all 50 samples, ranges from 20% to 40% and is much less in some samples (reaching 3%). The concentration percentage of diacetylmorphine in Kuwait is much less than that in Afghanistan (80%).

All heroin samples seized in Kuwait contain Papaverine (Papa) and Acetylcodaine (A-cod), with various concentrations, which are considered as derivatives of opium. They also contain 6-monoacetylmorphine (6-MAM) with low concentrations, as derivatives of heroin during the preparation by acetylation process, while morphine (Mor) has not been detected in all fifty heroin samples.

The most common additives detected in heroin samples are paracetamol or acetaminophen (Para) with percentage concentration ranging (0-21%) and caffeine (Caf) with percentage concentration ranging (0.5-16%). Other additives recorded in the samples include diazepam (Dia), Phenobarbital (Phe) and Alprazolam (Alpra), which are considered as important additives because of their drastic impacts which are more dangerous to human health than that of heroin, while diazepam and phenobarbital have the same effect of heroin but are more dangerous to human health than it. This would explain the

increasing number of deaths in Kuwait in the last few years of addicts taking heroin [15].

The similarity between the additives identified in heroin under study and those recorded in heroin manufactured in Afghanistan and Iran would suggest that the mixed heroin in the two countries are possible sources of heroin seized in Kuwait.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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