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Master Gas Chromatographical Identification of Condom Lubricants

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ABSTRACT

In most of the cases of sexual assault, scientists are in search of biological evidences like blood, skin, hair, saliva, urine, semen for DNA analysis. The uses of condoms in sexual assault cases were now increasing to avoid leaving the biological traces in the crime scene. In these cases, finding of the condom lubricant traces from victims can play a vital role in sexual assault cases. It has been found that gas chromatography gives an important method of such identifications. Gas chromatography was one of the important analytical tools used in majority of forensic laboratories. In present study condoms were referred to forensic science laboratories from police station was used to analyze these lubricants. The finding shows that majority of condom lubricants used were polydimethylsiloxane (PDMS). The Gas chromatograph spectra database of lubricants can be used to provide associative evidences between victim and sexual assailants.

Keywords: Gas chromatography, Condom lubricant and victim.

I. INTRODUCTION

In cases of sexual assaults, it is often found that the physical evidences from the victim are used for DNA analysis to obtain some evidences. Recently due to increasing public awareness, the offender often wore a condom to avoid the proof of biological evidences at the crime scene [1-6]. A vaginal swab may be taken from the victim to examine the condom lubricants even though the small amount of the lubricants can be found for analysis purpose [4, 7]. By analyzing the condom lubricants, one can obtain the valuable intelligence information between the victim and the suspect. Such trace evidences associated with the use of a condom can originate from the condom or additional use of lubricants. The components of the lubricants or additives used on condoms available in different brands are considerably different [5]. Condoms are manufactured from several materials, primarily synthetic polymers and sheep caecum. But the majority of condoms are made from latex rubber, which has the chemical structure *cis*-1,4-polyisoprene [8,9]. The major components can be used as the lubricants on condoms, such as polydimethylsiloxane (PDMS), polyethylene glycol (PEG), polypylene glycol (PG), and glycerin, though PDMS is far more common [5,10]. Some type of condoms may contain additives that give the lubricant formula with specific desired properties, such as its color, flavors, and perfumes [5, 10, 11]. In this study, we found that the oil lubricants and non-spermicidal condoms. Previous researches had used several techniques to detect lubricants on condoms, such as FTIR, high performance liquid chromatography (HPLC), NMR spectroscopy, Matrix-assisted laser desorption or Ionization-time of flight-mass spectrometry (MALDITOF- MS), Capillary electrophoresis, PyGC-MS, GCMS, and Raman spectroscopy [12]. Maynard et al. provided that the extraction and analytical protocol for lubricant analysis purpose from cotton swab. They showed that the infrared spectra may reveal the major type of the lubricants. For further discrimination of the lubricants or confirmatory identification test, PyGC-MS, fluorescence microscopy and fluorescence spectroscopy can be used as one of the technique [1]. Condom lubricants have been analyzed by using capillary electrophoresis (CE) by Burger et al. They showed that CE can detect and discriminate condom lubricants; however this instrument is not much common in forensic science laboratories. The limitation of this technique is that CE cannot identify lubricant residues correctly after lubricant 34 Forensic Science Journal 2012; Vol. 11, No. 1 recovery from the skin and cloth surfaces taken after 30min [5]. Lee et al. showed that NMR is a method capable of distinguishing between sexual lubricants used

in condoms by the different manufacturers. They developed the flow chart for differentiation of condoms using NMR method that could be easily implemented in any forensic laboratories [8]. However, the sample preparation and analysis time for NMR is time more and time consuming. NMR analytical method may not ideal for trace detection purpose [10]. In Hollenbeck et al.'s research, LC/ESI-MS, nano ESI-MS and MALDI-FTMS have been shown to be capable for identifying the traces of the spermicide nonoxynol-9 from internal vaginal swabs taken postcoitus, and in an actual evidence sample [13]. Spencer et al. had clearly shown that MALDI-TOF-MS is ideal for detecting condom lubricants and additives, especially the trace evidences were condom and personal lubricant residues and their mixtures with biological fluids. This research also used ATR-IR spectra to find the additives to the lubricant formula that were either undetectable or poorly detected in the mass spectrometer. ATR-IR provided valuable information about the additives found in PDMS-based lubricants. The present task showed that Infrared spectroscopy can used to support and clarify the MALDI data [10]. In recent year, gas chromatograph becomes the most popular instrument in the forensic science laboratory. Gas chromatographic analysis is the standard method for routine screening of swabs recovered from the sexual assault with respect to lubricants identification [2, 3, 12, 14]. Blackledge et al. had developed a protocol for the recovery of latex condom lubricants traces and their identification. FTIR was used to identify the PDMS and nonoxynol-9. They also used the desorption chemical ionization mass spectrometry (DCI-MS) to compare the PDMS from the different manufactures, the amount of sample size can reduced to 20mg of the material. PDMS tend to remain in the vaginal vault and had been successfully recovered and identified up to 24 hours after sexual intercourse [2,3]. In another Blackledge's research, they measured the viscosity by using Fourier selfdeconvolution (FSD) method to resolve overlapping IR spectral bands. They were successful in determining the average chain length of different PDMS polymers. FTIR with FSD could also be used as a preliminary screening procedure to identify PDMS traces and to determine the approximate viscosity [14]. Raman spectroscopy is a one of the complementary technique of FTIR. Coyle and Anwar have shown that Raman spectroscopy is an excellent tool for the screening of swabs for condom lubricants prior to DNA analysis. They found the majority of condoms were PDMS lubricants on the UK market [15]. Forensic scientists prefer using nondestructive methods of analyzing trace evidences rather than using destructive methods. Image documentation spectroscopy is a spectroscopic technique used for the nondestructive identification of molecular species, including polymer. In the forensic laboratory, trace evidences such as explosives, fibers, paints, pigments, drugs, inks, gunshot residues and forgeries and fakes can be analyzed by Image documentation. Gas chromatogram technique in the forensic science is sensitive enough for to detect the traces of the condom lubricants in extracts from cotton swabs. The purpose of present investigation is to use Gas chromatogram analyzing the condom lubricants associated with crime exhibits. Hopefully, the Gas chromatogram and image documentation database of the condom lubricants can give the information and spectral comparison for the forensic laboratories.

Chemicals and Reagents:

The chemicals Ether, used for analysis was HPLC grade and milli Q ultra pure water is used.

Preparation of sample Solution:

Cotton swabbing of condom was recovered from suspected accuse and recovered from crime scene were taken and extracted with 10ml of ether and further extract were concentrated to 1ml and injected on gas chromatogram for analysis.

II. Methods and Material

Instrumentation:

Gas chromatography

(A) The Gas liquid chromatography (DANI MASTER Gas Chromatograph) coupled with FID

(B) Operating Conditions: The following operating parameters were used

Column (Capillary): Dani–DN-5 MS capillary column (5% phenyl) - 95% Methyl polysiloxane Film thickness is 0.25µm, max temperature used 350ºC Non polar bonded and cross-linked, inertness low bleeding and of Good thermal stability

Column Material: The Capillary Length of 30mm, O.D 0.25mm, I.D. 0.25mm

Carrier Gas : Nitrogen, with flow rate 30ml/min.

Fuel : Hydrogen, flow rate 35ml/ min.

Air : Flow rate 350ml/min.

Split flow : 1:50

(C) Programming

A. Oven initial Temperature used is 240°C.
B. hold for 2 min
C. Heating Rate is 20°C
D. Oven final Temperature is 280°C
E. Injector Temperature is 280°C
F. Detector Temperature is 285°C

Image documentation : Make – Aetron

III. Results and discussions

Identification of Condom Lubricants by using the DANI master gas chromatograph technique, were done by using the samples prepared as per sample preparation were injected on gas chromatogram. The modular design of DANI master gas chromatograph components allows easy changing any GC configurations. Three injections units and three detectors units were mounted simultaneously. Dani selective detectors specifically eliminate the matrix interferences while providing the maximum sensitivity. The detectors data acquisition rate used is up to 300Hz for a better reproducibility and accuracy of chromatographic results. The Gas sampling valves, auxiliary ovens, switching valves and other optional devices make DANI MASTER Gas Chromatography as the most suitable gas chromatograph for the development of complex analytical systems in a wide range of applications. DANI MASTER GC can be connected to DDS CLARITY work station through a local area network (LAN). Dedicated turnkey systems are the added value of all DANI instruments. DANI MASTER GC coupled with DANI HSS 86.50 Head space sampler and DANI TD Thermal desorber, covers a wide range of applications for the environmental, chemical petrochemical pharmaceutical, food and beverages.

On condoms three types of lubricants were found and are polydimethyl siloxane (PDMS) polyethylene glycol (PEG) and glycerin, the majority condom lubricants were PDMS-based lubricants. Figure 1shows gas chromatogram of condom seized from accuses. Figure 2 shows gas chromatogram of condom seized from accuses. Even though some of the condoms indicated special favors, scents and colors, gas chromatogram can indicates the differences between them. If we overlay the gas of we can definitely conclude weather these are of same or not. However, gas chromatogram is a good and sensitive tool to detect the very minar amount of evidences.

Methods Applied for Detection of semen in condom are Classified as: [16]

1. Physical Examination
2. Chemical Examination
3. Microscopic Examination

Physical Examination
This includes the visual Examination. To naked eye seminal stains generally appear translucent or opaque spots, at times with yellowish tint and darker border depending on colour and thickness of substrata, which, if absorbent, also acquire stiffness due to dried semen. On good substrata seminal stains may appear to be fluorescent under the ultraviolet light.

Chemical examination:PH = 7.4 Alkaline.
The tests used to detect Seminal Stains are:

1. Florence Test
2. Barberio Test
3. Acid Phosphatase Test

MICROSCOPIC EXAMINATION:

The Microscopic detection of the seminal stains is based on morphology of spermatozoa. All above methods were applied for the detection of semen stain. It gives positive presence of semen in condom found at crime scene. Individualization of semen stains with help of Absorption elution method and cross over the electrophoresis technique.

Table remaining

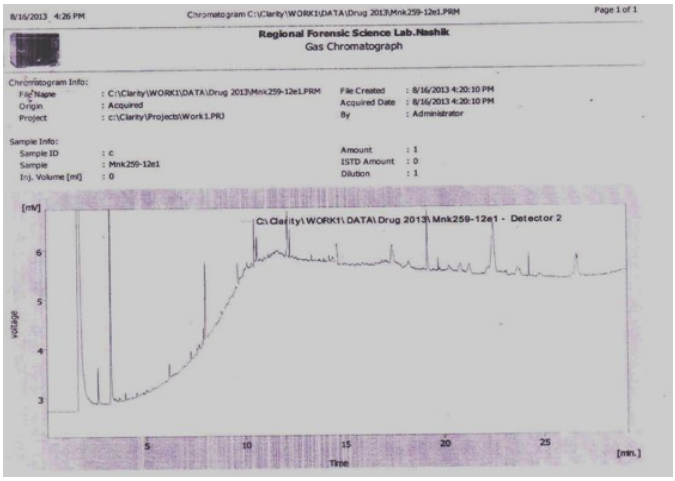

Figure 1. Gas chromatogram of condom seized from accuses.

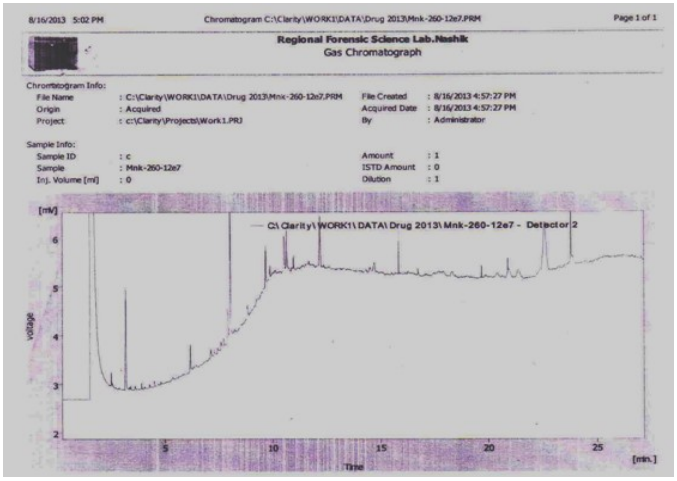

Figure 2. Gas chromatogram of condom seized from crime scene.

Figure 3. Spectrum taken on image documentation in top visible and trans visible mode.

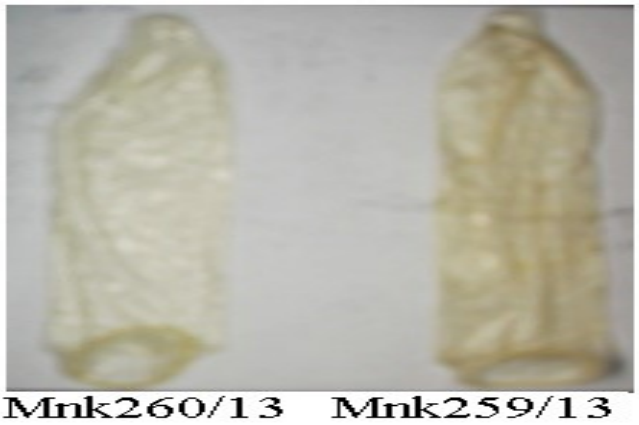

Figure 4. Spectrum taken on image documentation in top visible and trans ultra violet mode

Figure 5. Spectrum taken on image documentation in top UV and trans ultra violet mode.

IV. Conclusions

Condom lubricants have very good gas chromatogram and they can be identified with the help of gas chromatography. The Gas chromatogram is available in most of the forensic laboratories but it may not be sufficient to distinguish the polymers having the similar structures. Image documentation has shown that it is the most useful technique to analyze condoms as well as trace evidences collected for analysis in the forensic laboratories. This preliminary study gives a basis for further research to develop other good techniques for classification of condom lubricants in future.

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