

DETERMINATION OF PESTICIDE RESIDUE IN DIFFERENT HERBAL FORMULATION BY GAS CHROMATOGRAPHY

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ABSTRACT

Standardization of Ayurvedic & Herbal formulation is necessary to confirm its identity and to determine its quality, purity safety, effectiveness, and acceptability of the product that carried out by determination of Ash value, Extractive value, LOD, physical parameters which include Hardness, Friability, Disintegration time, dissolution time and HPTLC fingerprinting. pesticide residue in marketed hepatoprotective herbal formulation it was observed that the pesticide residues in formulations were present as in HP1 (Alpha HCH - 0.19ppm), HP2 (diazinone-0.63ppm), HP3 (Captafol-0.39ppm, Trans Fluthrin-0.27ppm), HP4 (Beta-HCH-0.20ppm, Heptachlor -0.38ppm), HP5 (Alachlor-0.27ppm) and H1 (Beta-HCH-0.42ppm), H2 (Endosulfan Sulphate-0.70ppm), H3 (Methoxychlor-0.18ppm), H4 (Endrin Ketone-0.12ppm), H5 (Alpha bhc-0.10ppm) which could produce ill effect on health of patient, using this herbal formulation hence it is necessary to analogue.

KEYWORDS: Ash Value, Herbal formulation, hepatoprotective.

INTRODUCTION

The WHO defines an herb as being fresh or dried, fragmented or powdered plantmaterial, which can be used in this crude state or further processed and formulated to become the final herbal product. Treatment of herbs by squeezing, steaming, roasting, decocting orinfusing in water, extracting with alcohol, or sweetening and baking with honey can create“herbal products” such as juices, tinctures, decoctions, infusions, gums, fixed oils, essentialoils, and

resins. The practices of most ethnic herbal medicine include the use crude or raw herbs that are collected from the wild or from cultivated fields and their prepared or ready-made (formulated mixture of herbal or other natural materials) products Toxic contaminants may come from Environments and conditions that the medicinal plants are grown or collected The conditions under which they are dried and processed, the storage conditions and conditions during transport. The manufacturing processes when the ready-made medicinal products are produced. WHO and FAO (Food and Agricultural Organization) set limits of pesticides, which are usually present in the herbs. These pesticides are mixed with the herbs during the time of cultivation. Mainly pesticides like DDT, BHC, toxaphene, aldrin, and endo sulfan cause serious side-effects in human beings if the crude drugs are mixed with these agents.

- Microbial contamination – usually medicinal plants containing bacteria and molds are coming from soil and atmosphere. Analysis of the limits of *E. coli* and molds clearly throws light towards the harvesting and production practices.

WHO, (1998) mentions maximum **permissible limits** in raw materials only for **arsenic, cadmium, and lead**, which amount to **1.0, 0.3, and 10 ppm**, respectively. The concentration of heavy metals is one of the criteria that make raw plants admissible to the production of medicines due to the fact that amount taken increases with the concentration, increased by constant mass of a taken dose.

One of the most common types of mass analyzer in GC/MS is the quadrupole ion-trap analyzer, which allows gaseous anions or cations to be held for long periods of time by electric and magnetic fields. A simple quadrupole ion-trap consists of a hollow ring electrode with two grounded end-cap electrodes as seen in figure #. Ions are allowed into the cavity through a grid in the upper end cap. A variable radio-frequency is applied to the ring electrode and ions with an appropriate m/z value orbit around the cavity. As the radio-frequency is increased linearly, ions of a stable m/z value are ejected by mass-selective ejection in order of mass. Ions that are too heavy or too light are destabilized and their charge is neutralized upon collision with the ring electrode wall. Emitted ions then strike an electron multiplier which converts the detected ions into an electrical signal. This electrical signal is then picked up by the computer through various programs. As an end result, a chromatogram is produced representing the m/z ratio versus the abundance of the sample.

DRUG PROFILE:**ANTIOXIDANT:**

Herbal churns containing *Withania somniferous* (Ashwagandha)

Hepatoprotective drug:

This mainly contains the following constituents in it which are as follows:

Himsra, Kasni, Mandur Bhasma, Kaka Machi (Solanum Nigrum), Arjuna (Terminalia Arjuna), Kasamarda (Cassia Occidentalis), Brinjasipha (Achillea Millefolium) & Jhavuka (Tamarix Gallica)

RESULT AND DISCUSSION**1. Organoleptic parameters:****Result for hepatoprotective****Table no 1: Organoleptic parameters result for hepatoprotective.**

<i>Sr. no</i>	<i>Taste</i>	<i>Odour</i>	<i>Colour</i>	<i>Appearance</i>
HP 1	Sweetish bitter	Aromatic	Grayish black	Red colored sugarcoated Tablet
HP 2	Bitter	Aromatic	Grayish white	Violet colored Tablet
HP 3	Sweetish bitter	Pungent	Grayish black	Pink colored sugarcoated Tablet
HP 4	Bitter	Pungent	Grayish black	Dark Pink colored Tablet
HP 5	Bitter	Aromatic	Grayish white	Reddish colored Tablet

Result for Antioxidants**Table no 2: Organoleptic parameters result for Antioxidant.**

<i>Sr. no</i>	<i>Taste</i>	<i>Odour</i>	<i>Colour</i>	<i>Appearance</i>
H 1	Tasteless	Odourless	Cream color	Cream colored powder
H 2	Tasteless	Odourless	Cream color	Cream colored powder
H 3	Tasteless	Odourless	Cream color	Cream colored powder
H 4	Bitter	Aromatic	Cream color	Cream colored powder
H 5	Bitter	Aromatic	Cream color	Cream colored powder

2) Result for Physiochemical parameters:**I. Determination of Ash Value****Hepatoprotective:****2.1 Determination of acid Insoluble Ash:****Table no.10: Physiochemical parameter result for hepatoprotective acid insoluble ash.**

Herbal Product Formulation Code	Acid Insoluble Ash	Mean \pm Standard deviation	% Mean
HP 1	0.03 0.02 0.03	± 0.05774	0.88

HP 2	0.04 0.02 0.02	0.02666 ±0.011547	0.88
HP 3	0.03 0.02 0.02	0.02333 ±0.05774	0.77
HP 4	0.02 0.03 0.03	0.02666 ±0.05774	0.88
HP 5	0.04 0.03 0.01	0.02666 ±0.011275	0.88

2.2 Determination of Water soluble Ash:

Herbal Product Formulation Code	Water Soluble Ash	Mean ±Standard deviation	% Mean
HP 1	0.02 0.05 0.01	0.02666 ±0.020817	0.88
HP 2	0.01 0.01 0.01	0.01 ±0	0.33
HP 3	0.05 0.04 0.03	0.04 ±0.01	1.33
HP 4	0.02 0.05 0.05	0.04 ±0.017321	1.33
HP 5	0.01 0.02 0.02	0.01666 ±0.005773	0.33
Herbal Product Formulation Code	Acid Insoluble Ash	Mean ± Standard deviation	% Mean
H 1	0.011 0.013 0.015	0.013 ±0.002	0.43
H 2	0.01 0.01 0.02	0.01333 ±0.015275	0.43
H 3	0.02 0.03 0.05	0.03333 ±0.015275	1.11
H 4	0.01 0.02 0.02	0.01666 ±0.005773	0.55
H 5	0.01 0.01 0.01	0.01 ±0	0.33

Herbal Product Code	Formulation	Water Soluble Ash	Mean \pm Standard deviation	% Mean
H 1		0.02 0.01 0.01	0.01333 \pm 0.005774	0.44
H 2		0.01 0.02 0.04	0.02333 \pm 0.0155	0.77
H 3		0.03 0.02 0.01	0.02 \pm 0.01	0.66
H 4		0.02 0.05 0.04	0.03666 \pm 0.015275	1.22
H 5		0.01 0.03 0.06	0.03333 \pm 0.025166	1.11

II. Determination of P.H:

Hepatoprotective:

Table no.14: Physiochemical parameter result for Hepatoprotective P.H.

Herbal Formulation code	Herbal Formulation 0.02 gm	Mean \pm Standard deviation	Herbal Formulation 2 gm	Mean \pm Standard deviation
HP 1	6.52 6.50 6.52	6.5133 \pm 0.011547	5.52 5.57 5.59	5.56 \pm 0.036056
HP 2	5.70 5.73 5.75	5.7266 \pm 0.025166	6.02 6.04 6.05	6.0366 \pm 0.015275
HP 3	7.02 7.05 7.03	7.0333 \pm 0.015275	5.59 5.55 5.57	5.57 \pm 0.02
HP 4	5.72 5.75 5.76	5.7433 \pm 0.020817	5.52 5.49 5.55	5.52 \pm 0.03
HP 5	7.02 7.04 7.06	7.04 \pm 0.02	6.05 6.08 6.09	6.0733 \pm 0.020817

Antioxidant:**Table no.15: Physiochemical parameter result for Antioxidant P.H.**

Herbal Formulation code	Herbal Formulation 0.02 gm	Mean Standard deviation ±	Herbal Formulation 2 gm	Mean Standard deviation ±
H 1	6.09 7.02 7.01	6.7066 ± 0.548574	5.62 5.61 5.61	5.61333 ± 0.005774
H 2	7.05 7.03 6.09	6.7233 ± 0.548574	6.61 6.72 6.75	6.6993 ± 0.73711
H 3	6.61 6.65 6.63	6.63 ± 0.02	7.03 7.09 7.05	7.0566 ± 0.030551
H 4	6.49 6.45 6.43	6.4566 ± 0.030551	5.43 5.48 5.45	5.4533 ± 0.025166
H 5	6.54 6.55 6.58	6.5566 ± 0.020817	5.44 5.48 5.45	5.4566 ± 0.020817

III. Alcohol Soluble Extractive Value:**Hepatoprotective:****Table no.16: Physiochemical parameter result for Hepatoprotective Alcohol Soluble Extractive Value.**

Herbal Product Formulation Code	Wt of Petridish	Wt of petridish + Residue	Actual weight	Mean + standard deviation	% Mean
HP 1	52.20 54.10 52.25	52.24 54.15 52.28	0.04 0.05 0.03	0.04 ± 0.01	0.8
HP 2	56.06 58.07 51.02	56.08 58.09 51.05	0.02 0.02 0.03	0.023±0.00577	0.46
HP 3	26.31 30.03 34.10	26.34 30.07 34.12	0.03 0.04 0.02	0.03 ± 0.01	0.6
HP 4	26.54 28.72 25.15	26.56 28.74 25.18	0.02 0.02 0.03	0.023 ± 0.00577	0.46
HP 5	32.58 40.10 38.03	32.60 40.12 38.06	0.02 0.02 0.03	0.023 ± 0.00577	0.46

Herbal Product Formulation Code	Wt of Petridish	Wt of petridish + Residue	Actual weight	Mean + standard deviation	% Mean
H 1	23.73 23.72 25.70	23.76 23.74 25.73	0.03 0.02 0.03	0.026 ± 0.00577	0.52
H 2	53.70 53.73 50.62	53.73 53.76 50.63	0.03 0.03 0.01	0.0233 ± 0.0115	0.46
H 3	76.06 73.04 72.12	76.08 73.09 72.13	0.02 0.05 0.01	0.0266 ± 0.0208	0.52
H 4	77.04 76.10 72.13	77.06 76.13 72.15	0.02 0.03 0.02	0.0233 ± 0.0115	0.46
H 5	83.39 80.12 76.68	83.40 80.15 76.70	0.01 0.03 0.02	0.02 ± 0.01	0.4

Determination of pesticide residue in herbal formulation of hepatoprotective:

Table No.20: Organochlorine Pesticide residue parameter result for Hepatoprotective.

Herbal Product Formulation Code	Pesticide Residue Content	Result	Detection limit in
HP 1	Alpha-HCH	0.19 ppm	0.1 ppm
HP 2	Diazinone	0.63 ppm	0.1 ppm
HP 3	Captafol Trans Fluthrin	0.39 ppm 0.27 ppm	0.1 ppm
HP 4	Beta-HCH. Heptachlor	0.20 ppm 0.38 ppm	0.1 ppm
HP 5	Alachlor	0.27 ppm	0.1 ppm

Determination of pesticides residue in herbal formulation of Antioxidant:

Table no.21: Organochlorine Pesticide residue parameter result for Antioxidant.

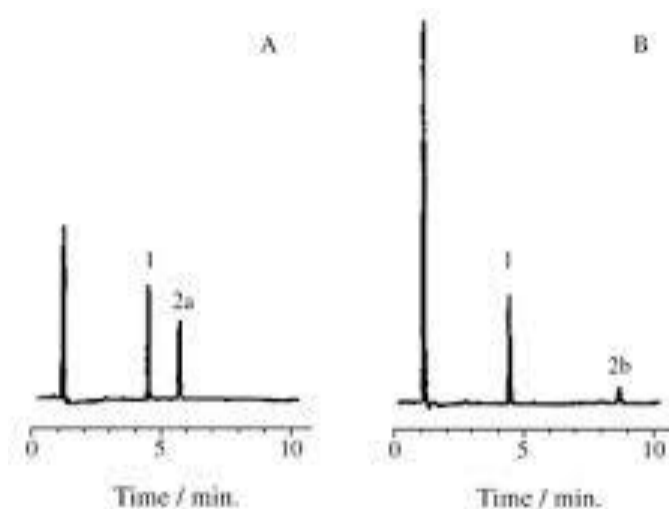
Herbal Product Formulation Code	Pesticide Residue Content	Result	Detection limit in
H 1	Beta-HCH	0.42 ppm	0.1 ppm
H 2	Endosulfan sulphate	0.70	0.1 ppm

		ppm	
H 3	Methoxychlor	0.18 ppm	0.1 ppm
H 4	Endrin ketone	0.12 ppm	0.1 ppm
H 5	Alpha bhc	0.10 ppm	0.1 ppm

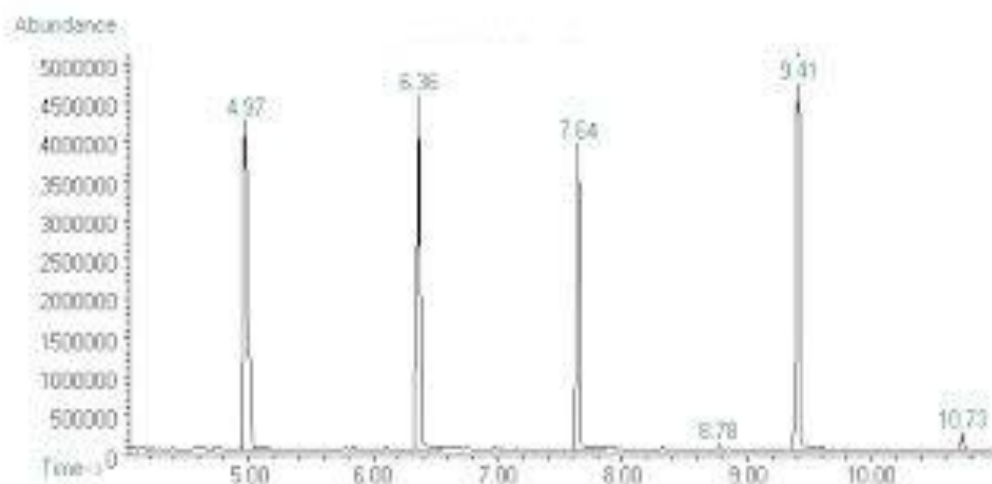
Organ chlorine graphs:

Hepatoprotective:

1 & 2: HP 1, HP 2



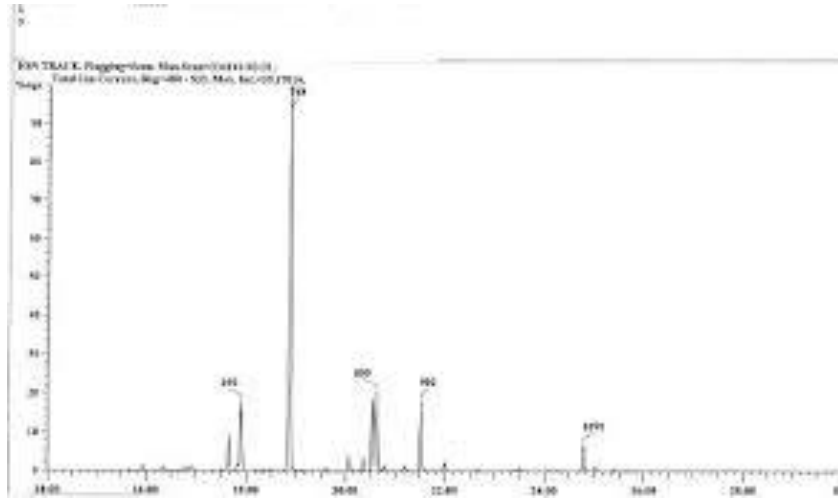
HP 3



:ALDRIN

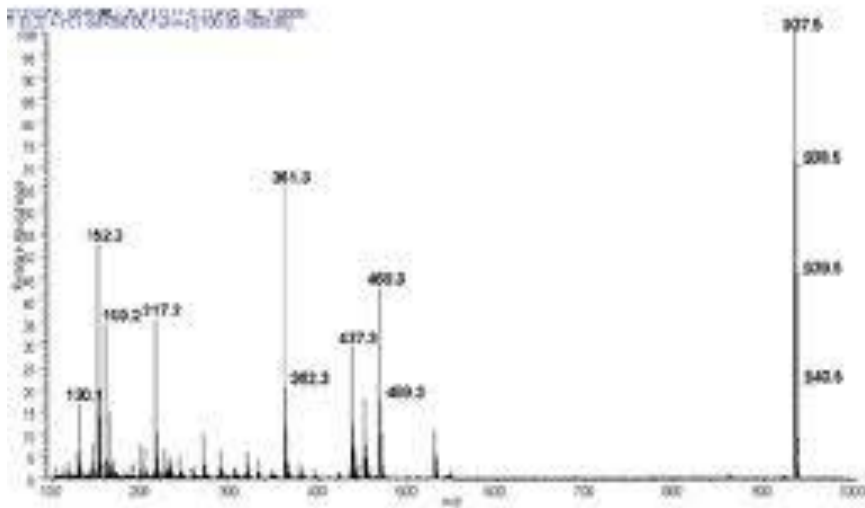
Anti oxidant:

1. H 1



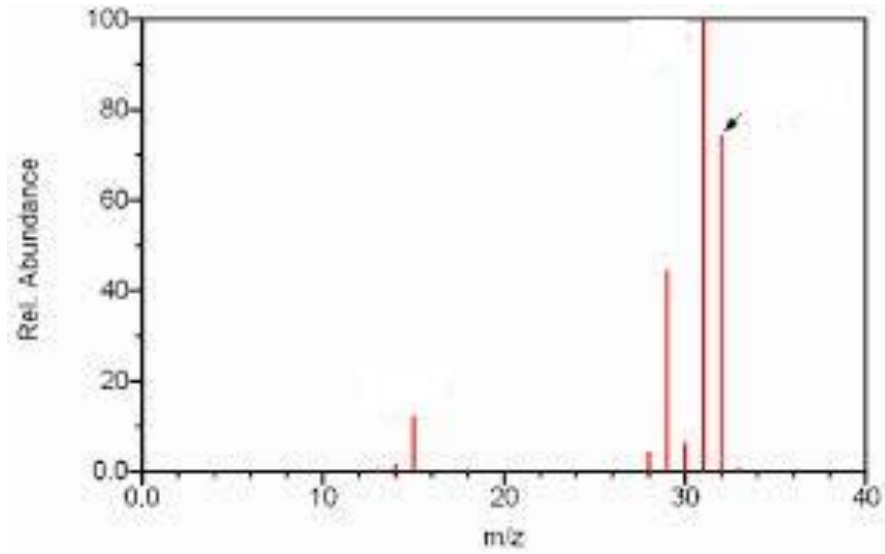
BETA: BHC

2. H 2



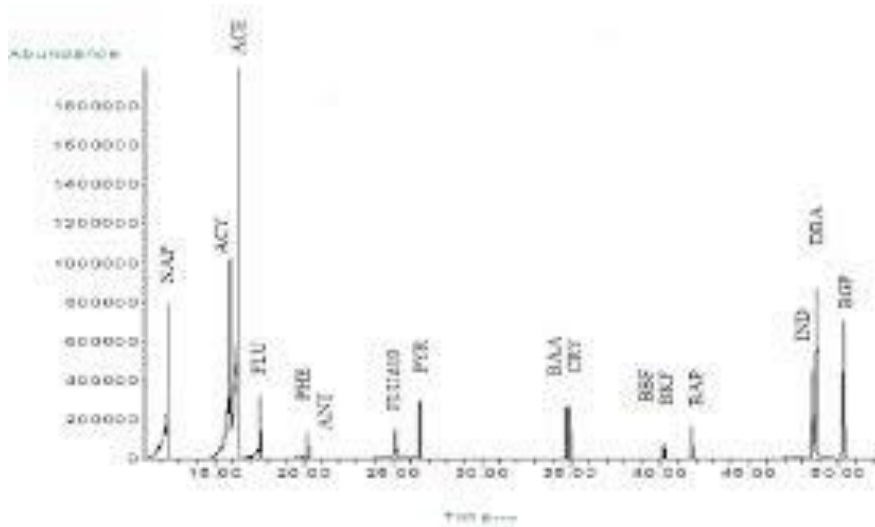
ENDOSULFAN SULFATE

3. H 3



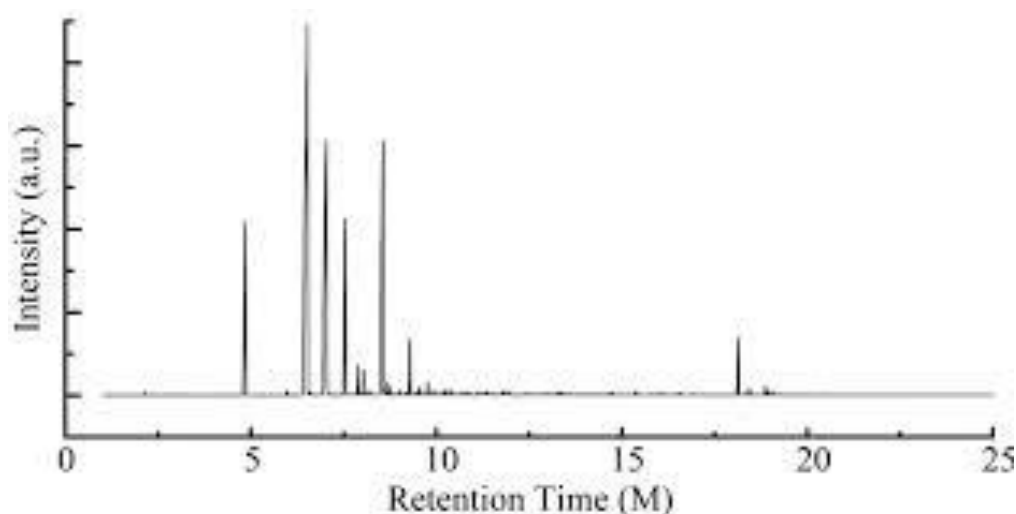
METHOXYCHLOR

4. H 4



ENDRIN KETONE

5. H 5



DISCUSSION:

From the result of determination of pesticide residue in marketed hepatoprotective herbal formulation it was observed that the pesticide residues in formulations were present as in HP1 (Alpha HCH -0.19ppm), HP2 (diazinone-0.63ppm), HP3 (Captafol-0.39ppm, Trans Fluthrin-0.27ppm), HP4 (Beta-HCH-0.20ppm, Heptachlor -0.38ppm), HP5 (Alachlor-0.27ppm).which could produce ill effect on health of patient, using this herbal formulation hence it is necessary to analogue.

From the result of determination of pesticide residue in marketed antioxidant herbal formulation it was observed that the pesticide residue in formulations were present as inH1 (Beta-HCH-0.42ppm), H2 (Endosulfan Sulphate-0.70ppm), H3 (Methoxychlor-0.18ppm), H4 (Endrin Ketone-0.12ppm), H5 (Alpha bhc-0.10ppm).which could produce ill effect on health of patient, using this herbal formulation hence it is necessary to analogue.

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