How to Cite:
Goel, K., Singh, R., Saini, V., & Sharma, M. (2022). Physicochemical evaluation of Solanum nigrum linn. and Tribulus terrestris linn. *International Journal of Health Sciences*, *6*(S4), 10786–10795. https://doi.org/10.53730/ijhs.v6nS4.10898

Physicochemical evaluation of Solanum nigrum linn. and Tribulus terrestris linn.

Kirti Goel
Research Scholar, Maharishi Markandeshwar College of Pharmacy, MM (DU), Mullana, Ambala, Haryana, India
Email: kirtijain2727@gmail.com

Randhir Singh
Associate Professor, Central University of Punjab, Bhatinda, Punjab, India
Email: randhirsingh.dahiya@gmail.com

Vipin Saini
Director, RAAAC, Maharishi Markandeshwar College of Pharmacy, MM (DU), Mullana, Ambala, Haryana, India
Email: vipinsaini31@rediffmail.com

Manu Sharma
Professor, Maharishi Markandeshwar College of Pharmacy, MM (DU), Mullana, Ambala, Haryana, India
Corresponding author email: lantadene@mmumullana.org

***Abstract*---**Herbal drugs are an integral part Indian medicinal system. Among them two plants are extensively used in folklore medicine system viz. Tribulus terrestris and Solanum nigrum because of numerous pharmacological activities. Various species of these plants are available, out of which some species are more potent whereas others have very less pharmacological potential. Therefore, identification of appropriate plant species is very important in the process of drug discovery and development. In the present study various physicochemical parameters were employed to identify powdered samples of fruit of Tribulus terrestris and berries of Solanum nigrum. These parameters include colour, odour, and taste. The physicochemical parameters evaluated for Tribulus terrestris and Solanum nigrum were found as moisture content (4.81, 6.89 respectively), loss on drying (5.16, 11.18 respectively) total ash values (7.44, 9.21 respectively) water soluble ash (4.081, 6.89 respectively), acid insoluble ash (0.81, 0.58) bulk density (0.50, 0.71) and tapped density (0.42, 0.65). The preliminary estimation of phytoconstituents were done by using TLC with

different solvent systems and the Rf values were compared with standard. The λmax obtained for Tribulus terrestris and Solanum nigrum was found to be 201nm and 267 nm respectively.

***Keywords*---**Tribulus terrestris, Solanum nigrum, authentication, physicochemical properties.

Introduction

Plants were always an integral part of medicinal system all over the world including Unani, Chinese and Ayurveda due to their medicinal and nutritional properties (Annan K, Houghton PJ 2008). The traditional Indian medicine system employs a myriad of flora due to their ease of availability in local ambience. The therapeutic efficacy of these plants depends upon the purity of phytoconstituents. The composition of phytoconstituents varies in different species of same plants and hence the qualitative and quantitative assessment is considered as a critical parameter in identification of medicinal plants (Raskin I et al 2002).

Physico-chemical assessment of crude drug involves validation of its identity and determination of its quality, purity and determination of adulteration. The evaluation of crude drug is essential due to 2 major factors i) Disparity of phytoconstituents in the drugs ii) Substitution and adulteration. Over the years, the nature and degree of evaluation of crude drugs has undergone a systematic changes. Initially, the crude drugs were identified by comparison only with the standard description available (White PJ, Brown PH 2010). Presently, due to advancement in the knowledge and techniques, evaluation of crude drugs includes determination of active constituents present in the crude drug along with organoleptic analysis. Pharmacognostic evaluation is the initial step to confirm the identity and to assess the quality and purity of crude drug. With the advent of separation techniques and instrumental analysis, it is possible to perform physical evaluation of a crude drug, which could be both of qualitative and quantitative in nature. *Tribulus terrestris* Linn. & *Solanum nigrum* Linn. are among the prominent medicinal plants in Indian folklore medicinal system owing to their effectiveness in various ailments (William Omuketi Emitaro2017, Romana Rashid 2017). These two medicinal herbs has a number of many species, out of which some are therapeutically effective whereas few are therapeutically inactive. Selection of appropriate species is very important for the pharmacological activity of plants. The organoleptic and physicochemical parameter are considered as important parameters for determination of appropriate species (Atanu U 2010, Rauf A, Waseem M, Rehman S 2019).

Materials and Methods

Plant Collection and Authentication

The berries of *Solanum nigrum* Linn and fruits of *Tribulus terrestris* Linn were purchased from local market during their favourable season (Table 1). The authentication was carried out at Sri Vankateshwara University, India having voucher numbers 0869, 0540.

Table 1. Plant details

S.No	Plant species(Family)	Common name	Part of plant taken for activity	Collection month
1	*Tribulus terrestris* L. (Zygophyllaceae)	Gokhru	Fruit	June-December
2	*Solanum nigrum* L. (Solanaceae)	Makoi	Berries	September-Nov

Sample Preparation

The authenticated plant material was washed thoroughly under running tap water and shade dried. The dried berries of *Solanum nigrum* Linn and fruits of *Tribulus terrestris* Linn was ground into powder form. The powdered plant material was stored in air tight jars for evaluation purpose.

Estimation of Organoleptic and Physicochemical parameters

Organoleptic evaluation involves the assessment of colour, odour, taste and appearance of powdered plant samples. The physicochemical study comprises of evaluation of successive extractive value of powdered samples in different solvents, moisture content, total ash values, acid insoluble ash values, water soluble, and loss on drying, bulk density (Chhatre S 2014).

Extract Preparation

The 2 different methods were adopted for preparing various extracts of berries of *Solanum nigrum* Linn and fruits of *Tribulus terrestris* Linn viz. continuous sequential soxhlation and hot maceration with solvents like petroleum ether, chloroform, ethylacetate, hydro alcohol (40:60) and water maintaining temperature not more than 40^0C for 72 hours (Khandelwal K. R 2002, Rauf A, Waseem M , Rehman S 2019). The extracts were dried to calculate extractive values and stored in air tight container for further usage (Mukharjee PK 2008, Indian Herbal Pharmacopoeia 2002). The extractive values were calculated by using following equation -01:

$$\text{Extractive value \%} = \frac{\text{Wt. of dried extract}}{\text{Wt. of plant material}} \times 100 \quad \text{[eq.01]}$$

Moisture content Determination

The moisture content in the powdered sample of berries of *Solanum nigrum* Linn and fruits of *Tribulus terrestris* Linn was measured by using Karl Fischer apparatus. The methanol used in this method was freed from moisture content and 200mg of powdered form of both plants were used to measure moisture content. Initial and final readings of burette were recorded (Toshiyuki Osakai 2017). The moisture content in the sample was calculated by using following equation.02:

"% Moisture content=(K.F in ml ×MDF ×100)/(weight of the sample") [eq.02]
Where, MDF stands for Moisture Determining factor of
K.F=5mg/ml

Loss of weight on drying

In a petridish 20 gm of drug was taken and subjected to heating at a constant temperature of 105^0C afterwards cooled in a desiccator and weighed and repeated the process until constant weight. The process was repeated till two successive weights were persistent (Jenkins G.I 2008). The %w/w loss in weight was calculated by using equation 03 given below.

Total Ash:

$$\text{\%Loss on drying} = \frac{\text{Loss in weight of sample}}{\text{weight of sample}} \times 100 \quad \text{[eq.03]}$$

In a pre-weighed silica crucible, 2 gm of plant sample was ignited at temperature 800^0C in a muffle furnace till sample is carbon free then it was cooled down in desiccator at room temperature and weighed (Mukharjee PK 2008). The total ash percentage was estimated by using following equation 04.

$$\text{Total ash} = \frac{\text{Final wt. of crucible and sample after ignition}}{\text{Initial wt. of crucible and sample}} \times 100 \quad \text{[eq.04]}$$

Water Soluble Ash

The total ash achieved was boiled for 5 minutes with 25 ml water and the insoluble content was transferred on an ash less filter paper which was treated with hot water and incinerated in a crucible for 15 minutes at a temperature not more than 450^0C. The left residue was weighed and subtracted from the obtained total ash. The content of water soluble ash

was evaluated with reference to dried plant material (Mukharjee PK 2008).

Acid Insoluble Ash

The acquired total ash mixed with 25 ml of dil. hydrochloric acid was boiled for 5 minutes. In gauch crucible or an ash less filter paper insoluble content was transferred and washed with hot water followed by incineration to obtain constant weight. The content of acid insoluble ash was evaluated with reference to air dried plant material (Chase CR, Pratt R 1948).

Poured Bulk Density: 50 gm of plant material was poured into graduated cylinder and the volume occupied by the plant powder was recorded (Rajani M, Kanaki 2008, NS Lachman L *et al* 1991)
Tapped Bulk Density: 50 gm of powdered plant was transferred into a graduated cylinder (volume occupied recorded) and allowed tapping for 15 minutes on Bulk density-tapped density apparatus (Rajani M, Kanaki 2008, NS Lachman L *et al* 1991).

Bulk Density and tapped density was obtained by employing following equation 05, 06:

$$\text{Bulk density} = \frac{\text{Mass of the sample taken (gm)}}{\text{Initial volume of sample (ml)}}$$

[eq. 05]

$$\text{Tapped density} = \frac{\text{Mass of the sample taken (gm)}}{\text{Final tapped volume of sample (ml)}}$$

[eq.06]

UV-Spectroscopic overlay

The solution of 100 μg/mL of both the plant samples were prepared (ethylacetate and hydroalcoholic extract) and analysed in UV-1800 Schimadzu for λmax (Gitu Pandey, Brahmeshwar Mishra 2013).

Thin Layer Chromatography (TLC)

Thin layer chromatography of extracts of the both the plant extracts was carried out on TLC silica glass plates using different mobile phases and they were sprayed visualizing reagents for identification of spots on the developed TLC plates. The Rf values of the spots calculation was done by using the following equation 07 (Gupta MM, Sharma RK 1996, Zafar R, Lawani M 1989, Indhumathi.T, Dr. S. Mohandass 2013)

Result and Discussion

$$\text{Rf value} = \frac{\text{Distance travelled by spot}}{\text{Distance travelled by solvent}} \quad \text{[eq.07]}$$

Estimation of organoleptic and Physico-chemical parameters

The assessed organoleptic parameters for both the plant samples are summarized in table: 2(Organoleptic parameters evaluation) which depicted that Fruits of Tribulus terrestris are moderately coarse having greenish yellow colour with agreeable odour and bitter taste while the berries of Solanum nigrum was in fine powder form having brownish black colour with agreeable odour and bitter taste. The moisture content present in *Tribulus terrestris* and *Solanum nigrum was* found to be 4.0 and 10.27 respectively. Loss of weight on drying was found to be 5.16%w/w, 11.18%w/w for *Tribulus terrestris* and *Solanum nigrum* respectively.

The ash values for *Tribulus terrestris* and *Solanum nigrum*were found to be considerable total ash -7.44, 9.1, water soluble ash -4.81, 6.89, and acid soluble ash-0.81, 0.68 respectively. The tapped density was evaluated as 0.42, 0.65 while Bulk density was found to be 0.50, 0.71 for *Tribulus terrestris* and *Solanum nigrum* respectively. The results are provided in table 3. (Physicochemical parameters estimation).

Table 2. Organoleptic parameters evaluation

S.NO	PLANT	ORGANOLEPTIC PROPERTY			
		COLOUR	APPEARANCE	ODOUR	TASTE
1	*Tribulus terrestris* (fruit)	Greenish with a tint of yellow	Moderately Coarse	Agreeable	Bitter
2	*Solanum nigrum* (Berries)	Brownish black	Fine powder	Agreeable	Bitter

Table 3. Physicochemical parameters estimation

S.NO	PARAMETERS ASSESSED (% W/W)	PLANT	
		Tribulus terrestris (fruit)	*Solanum nigrum* (Berries)
1.	Total ash	7.44	9.21
2.	Acid insoluble ash	0.81	0.58
3.	Water soluble ash	4.81	6.89
4.	Moisture content	4.0	10.27
5.	Loss on drying	5.16	11.18
6.	Bulk density	0.50	0.71
7.	Tapped density	0.42	0.65

Extractive Values

The percentage yield for the extracts prepared by 2 different methods are given in table: 4 (Extractive values). The soxhlation method was found to be more productive using petroleum ether as solvent while hot maceration method was more productive with hydroalcoholic solvent for *Tribulus terrestris*. The percentage yield of *Solanum nigrum* berries in each solvent was relatively more than *Tribulus terrestris* fruits.

Table 4. Extractive values

	Soxhlation method yield(%w/w)		**Hot maceration method yield(%w/w)**	
Solvent	***Tribulus terrestris***	***Solanum nigrum***	***Tribulus terrestris***	***Solanum nigrum***
Petroleum ether	2.78	5.915	1.64	3.18

UV Spectroscopic Overlay

The figure 1. Shows the λmax obtained by UV spectroscopic method wherein SN-HA, SN-EA stands for *Solanum nigrum* hydroalcoholic extract and ethylacetate extract of 100ug/ml concentration respectively and λmax was found to be 267nm. The λmax for *Tribulus terrestris* fruit extracts (HA-hydroalcoholic, EA-ethylacetate) was found to be 201nm.

Thin Layer Chromatography (TLC)

The different solvent system used for TLC revealed different spots for both the plant extracts. With the usage of Toluene: Ethyl Acetate in the ratio of 14:1 for *Tribulus terrestris* extract it shows 2 spots having Rf value of 0.24, 0.52 and Toluene: Acetone: Methanol: NH3 (20: 20: 3: 1 v/v) system with visualizing agent Freshly prepared Dragendorff's reagent gives 1 spot of Rf value 0.38 while the solvent system of Methonol: acetic acid (9:1 v/v) showed 3 spots having Rf value of 0.48, 0.65, 0.84. These Rf values for phytoconstituents belongs for alkaloids and flavonoid class.
The solvent system Chloroform Methanol in the ratio 95:5 revealed 2 spots of Rf value 0.19, 0.25 for Solanum Nigrum extract. Two spot has been showed up in solvent system of Toluene: Acetone: Methanol: NH3 (20: 20: 3: 1 v/v) having Rf value of 0.27, 0.39 and with the solvent system Acetone Water: Conc. ammonia (90:7:3 v/v) showed one spot having Rf value of 0.42. The calculate Rf values for different spots belongs to flavonoid family.

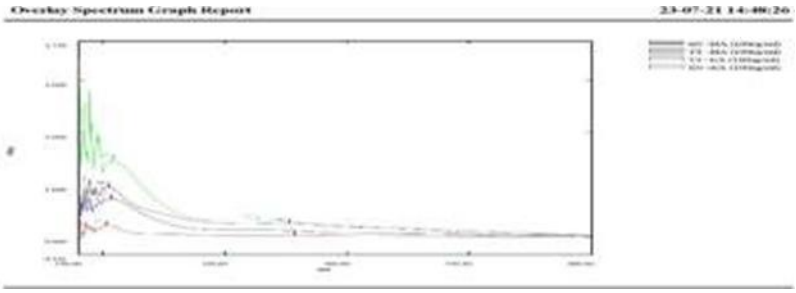

Figure 1. Overlay of Solanum nigrum berries and Tribulus terrestris fruits extracts

Table 5: Thin layer Chromatography of Tribulus terrestris and Solanum nigrum extracts

Plant extract	Solvent system	Visualising agent	No. of spots	Rf values
Tribulus terrestris				
1.	Toluene: Ethyl Acetate (14:1v/v)	Anisaldehyde-H_2SO_4 + Heating (100°C) for 10 min	2	0.24, 0.52
2.	Toluene: Acetone: Methanol–NH3 (20 : 20 : 3 : 1, v/v)	Freshly prepared Dragendorff's reagent	1	0.38
3.	Methanol: acetic acid (9:1/v/v)	Iodine chamber	3	0.48,0.65,0.84
Solanum nigrum				
1.	Chloroform Methanol 93:5	Freshly prepared Dragendorff's reagent	2	0.19, 0.25
2.	Toluene Ethyl acetate Methanol Formic acid 4.5: 5:6:1	Anisaldehyde-H_2SO_4 + Heating (1000C) for 10 min	2	0.27, 0.39
3.	Acetone Water Con ammonia 90:7:3	Dragendorff's reagent	1	0.42

Conclusion

The physicochemical properties of powdered samples of fruits of *Tribulus terrestris* and berries of *Solanum nigrum* are comparable with the standard specifications.

Table/figures list:

Sr.no	Table name/figure name
Table 1	Plant details
Table 2	Organoleptic parameters evaluation
Table 3	Physicochemical parameters estimation
Table 4	Extractive values
Table 5	Thin layer Chromatography of Tribulus terrestris and Solanum nigrum extracts
Fig.1	Overlay of Solanum nigrum berries and Tribulus terrestris fruits extracts

References:

Annan K, Houghton PJ (2008), "Antibacterial, antioxidant and fibroblast growth stimulation of aqueous extracts of Ficus asperifolia Miq. and Gossypium arboreum L", Journal of Ethnopharmacology .141-144.

Atanu U, Ebiloma, Ajayi EI, (2010) "A Review of the Pharmacological Aspects of *Solanum nigrum* Linn".

Bijauliya, R.K., Alok, S., Chanchal, D.K. and Kumar, M (2017). "A comprehensive review on Standardization of Herbal Drugs",International Journal of Pharmaceutical Sciences and Research, vol 8 no.9,pp. 3663-3667.

Biotechnology and molecular Biology Review vol 6, pp.1-7.

Chase CR, Pratt R (1948), "Fluorescence of powdered vegetable drug with particular reference to development of a system of identification", Journal of American Pharmaceutical Association 38, 324-331.

Chhatre S, Nesari T, Somani G, Kanchan D, Sathaye S. (2014) Phytopharmacological overview of Tribulus terrestris. Pharmacogn Rev.8(15):45-51. doi: 10.4103/0973-7847.125530.

Gitu Pandey, Brahmeshwar Mishra (2013), "A New Analytical Q-Absorbance Ratio Method Development and Validation for Simultaneous Estimation of Lamivudine and Isoniazid", Hindawi Publishing Corporation ISRN Spectroscopy. 1-5.

Gupta MM, Sharma RK, (1996) "Combined thin-layer chromatography densitometry method for quantitative determination of alkaloids of poppy straw samples", Indian J. Pharm. Sci., 161–164.

Indhumathi.T, Dr. S. Mohandass (2013), "Identification of bioactive compounds in *Solanum incanum* fruit by Thin layer Chromatography and HPTLC", International journal of Scientific Research, 2(6), 1-4.

Indian Herbal Pharmacopoeia, Indian Drug Manufacturers' Association. Revised ed. (2002). 493–4

Jenkins, G.I, Kenevel, A.M, and Degage, F.E. (2008), "Quantitative Pharmaceutical Chemistry", CBS Publishers & Distributors PVT. LTD., Daryaganj, New Delhi, Edn 6, 229-230, 239-242.

Khandelwal K. R, (2002) “Practical Pharmacognosy, Technique and Experiments”, Nirali Prakashan, Ninth Edition, 23.10-23.11 and 25.1-25.6.

Lachman L, Liberman HA, Kanig JL (1991),”The Theory and Practice of Industrial Pharmacy” Mumbai: Varghese Publishing House, 3rd ed. 67.

Mukharjee PK (2008)., “Quality Control of Herbal Drugs”, New Delhi: Business Horizons Pharmaceutical Publishers, 186,189.

Perveen S, Wadud A, Ahmed Makbul SA, Sofi G, Perveen (2019) A. Unani concept of drug substitution (therapeutic interchange) and its validation on scientific parameters. J Ayurveda Integr Med. doi: 10.1016/j.jaim.2017.11.006.

Rajani M, Kanaki NS (2008), “Phytochemical Standardization of Herbal Drugs and Polyherbal Formulations, Bioactive Molecules and Medicinal Plants”. Berlin, Heidelberg: Springer. 349–69.

Raskin I, Ribnicky DM, Komarnytsky S, Ilic N, Poulev A, Borisjuk N, Brinker A, Moreno DA, Ripoll C, Yakoby N, O'Neal JM, Cornwell T, Pastor I, Fridlender B (2002). Plants and human health in the twenty-first century. Trends Biotechnol. 20(12):522-31. doi: 10.1016/s0167-7799(02)02080-2. PMID: 12443874.

Rauf A, Waseem M , Rehman S. (2019) Physicochemical Standardization and Safety Evaluation of Khar-e-Khasak Khurd (Fruits of Tribulus terrestris): A Unani Drug. J Pharmacovig Drug Safety. 16(1):16-21.

Romana Rashid, Dr. Ghulam Nabi Hajam, Muzaffer Hussain Wani, Dr. Manik Sharma(2017). Organoleptic evaluation of the plant Solanum nigrum L. of the family Solanaceae. International Journal of Applied Research, vol 3(11) 142-145.

Suryasa, I. W., Rodríguez-Gámez, M., & Koldoris, T. (2021). Get vaccinated when it is your turn and follow the local guidelines. *International Journal of Health Sciences*, *5*(3), x-xv. https://doi.org/10.53730/ijhs.v5n3.2938

Toshiyuki Osakai (2017), “The Principle of Water-Content Determination by Karl Fischer Titration”. Review of Polarography, vol 63.101-107.

White PJ, Brown PH.(2010) Plant nutrition for sustainable development and global health. Ann Bot. 2010 Jun; 105(7):1073-80. doi: 10.1093/aob/mcq085. .

William Omuketi Emitaro, David Mutisya Musyimi, Darius Andika Otiato, Benson Onyango. 2017 Morphological, biochemical and molecular characterization of Xanthomonas campestris pv. vesicatoria of African nightshades (Solanum scabrum

Zafar R, Lawani M (1989), “*Tribulus terrestris* Linn. - A review of current knowledge”, Indian Drugs, 27(3).148-158.