The effect of different drying temperatures, phytochemical and nutrient retention in freeze dried Moringa flowers (*Moringa oleifera* L.)

G. Pandidurai[1,*], S. Amutha[2], S. Kanchana[3], S. Vellaikumar[4] and K. Prabhakaran[5]

[1,2&3] Department of Food Science and Nutrition, Community Science College and Research Institute, Tamil Nadu Agricultural University, Madurai

[4]Department of Biotechnology, Agricultural College and Research Institute, Madurai

[5]Department of Social Science, Agricultural College and Research Institute, Killikulam.

**Corresponding author. Email: nanalpandi@gmail.com*

Abstract

Moringa (Moringa oleifera Lam.) is an incredible plant to humans because of its pharmacognostic and nutraceutical properties. Moringa oleifera contains vital nutrients such as minerals, vitamins and phytochemicals such as tannins and flavonoids. The present work aims to study the optimization and quality attributes retention in moringa flower through freeze dryer (FD) because the conventional drying process takes more time and energy which also affects the product quality and safety. Different solvents, such as ethanol, chloroform, hexane, acetone and ethyl acetate, were used to determine the presence of phytochemicals (alkaloids, tannins, flavonoids, steroids, terpenoids and saponins) in the moringa flower. From Qualitative and quantitative screenings of moringa flower, the phytochemicals were highly present in methanol and ethanol extracts. Dried moringa flower powder was prepared by using fresh moringa flower, steam blanched for 2-5 min and then sulfated at 0.1 % for 10 min. After that, the dehydration process was performed at different temperatures (45, 55 and 65°C) in an FD drier. FD dried moringa flower powder at 55℃ was found maximum physicochemical properties and higher retention of phytochemical with special reference to β – carotene (55.41 mg), total antioxidant (460.18 mg), flavonoids (19.34 mg/RE), phenol (234.13 mg/GAE), vitamin C (485.09 mg).

Keywords: *Moringa flower, Freeze dryer, nutraceutical and phytochemicals.*

INTRODUCTION

Moringa oleifera (*M. oleifera*) is also known as the "tree of life" because of its crucial importance. Moringa belongs to the solitary genus from the family *Moringaceae* and contains 13 known species. Among them, *M. oleifera* is a highly exploited species. The other names used for moringa are horseradish tree, Mulangay, benzolive, drumstick tree, Sajna and kelor. It is also popularly denoted as a miracle tree", "natural gift or "mother's best friend" (Azevedo *et al.,* 2018). India is a leading moringa producer in the world, with a yearly production of 2.2 million tons and a productivity of 51 tons of tender pods per hectare. Tamil Nadu is the leading moringa-producing state in India, with an average production of 6.71 lakh tonnes of tender pods annually from a cultivated area of 13042 ha (Sekhar *et al.*, 2018). Moringa has been used in folk medicine to treat many diseases, including cardiac and circulatory stimulants; antitumor, antipyretic, antiepileptic, anti-inflammatory, antiulcer, antispasmodic, diuretic, antihypertensive, lowering cholesterol, antioxidant, antidiabetic, hepatoprotective, antibacterial, and antifungal properties; and antitumor, antipyretic, antiepileptic, anti-inflammatory (Anwar *et al.*, 2007). Furthermore, evidence points to antioxidant activity as one of the main mechanisms of action underlying the medicinal properties of moringa leaf (Shailendra *et al.*, 2016). To prevent postharvest losses in the moringa, there is a need for processing, and it will meet the demand of the market throughout the year (Aliyu *et al.*, 2016).

Dehydration is a traditional method of preservation; hot air is used to dry food and plant materials. During the drying process, a higher temperature can lower the flavour, colour, heat-sensitive nutrients and bioactive compounds. The quality of the dried products can be improved by reducing the process temperature when compared to higher ones. Based on the product type, availability of dryer, cost, time and energy consumption desired drying techniques (Kinki *et al.*, 2020). The conventional drying process takes more time and energy than advanced techniques, and it also results in microbial contamination of food products due to prolonged processing time and improper handling, which will also affect the organoleptic properties and product quality in terms of mould growth during storage. Alternatives to conventional drying advanced dryers, *viz.*, freeze- or vacuum-drying and heat pump-assisted dehumidified air drying (HPD), is used for dry heat-sensitive materials. (Klungboonkrong *e al.*, 2018).

Freeze drying

Because of the low temperature and lack of atmospheric air, freeze-drying preserves the natural product's constituents, allowing the powder's chemical, nutritional, and sensory properties to remain virtually unchanged, as well as having characteristics that inhibit the growth of microorganisms that could cause it to deteriorate (Silva *et al.*, 2019). Freeze-drying is commonly employed to preserve *M. oleifera* leaves in impoverished nations that grow and prepare them, although the high and often changing temperatures during post-harvest often have negative impacts on health-promoting bioactive components of foods (Barimah *et al.*, 2017). Lyophilization, also known as freeze-drying (FD), is a novel technique that involves lowering and maintaining the effective product temperature below 0°C while simultaneously applying reduced pressure (usually around 600–700 Pa) to ensure that all of the product's moisture is sublimated from the solid-state to the vapour state. Due to drying at sub-zero temperatures, which causes no thermal degradation and resists the operation of degradative enzymes by reducing moisture, FD is superior to conventional drying methods in terms of retaining active components of produce. (Klungboonkrong *et al.*, 2018).

Generally, moringa leaves are more focused than moringa flowers because of its high nutritional benefits and availability but the research on moringa flowers are unexplored. The present study aims to optimize the freeze-drying parameters of moringa flower powder.

MATERIALS AND METHODS

Physico-chemical properties of moringa flowers

A fresh and healthy PKM – 2 variety moringa flowers procured directly from a farmer producer organization in Madurai was used in the study. Fresh moringa flowers were analysed for their physicochemical characteristics, *viz.,* moisture and ascorbic acid, by the methods given by AOAC (1990) and Ranganna (1995). The protein-Kjeldahl method was used to measure total nitrogen content. Crude fibre (Abbas *et al.,* 2018), β – carotene – HPLC method (Chen *et al.,* 2017) total antioxidant activity (TAA) (Lim *et al.,* 2007), total flavonoids (Meda *et al.,* 2005), total phenols (TP) (Quettier-deleu *et al.,* 2000) and minerals were analysed by Boudieb *et al.,* 2019.

Phytochemical screening of fresh moringa flowers

Extraction of bioactive compounds from fresh moringa flowers was carried out using different solvents like methanol, ethanol, chloroform, hexane, acetone and ethyl acetate. 20 grams of the fresh moringa flower samples were taken into a 250 ml beaker. After that, 150 ml of the different solvents was added and centrifuged at 1000 rpm for 2 hr until agitation. Then, the samples were allowed to continue the extraction process at room temperature for 72 hr. Finally, each sample solvent mixture was filtered, and the crude extract was taken for phytochemical screening (Marcel *et al.,* 2016). Qualitative phytochemical analysis was performed with each solvent extract of the moringa flowers to determine the presence of alkaloids, tannins, flavonoids, steroids, terpenoids and saponins **(**Andzouana and Mombouli 2012).

Processing for the preparation of dried moringa flower powder (MFP)

Healthy mature moringa flowers were selected, and then fresh moringa flowers were steam blanched for 2-5 min until tender and sulphated at 0.1 % for 10 min to preserve the colour and to improve storage durability. After that, a dehydration process was performed with freeze-drying (Silva *et al.,* 2019). A Liotop® L101 tabletop freeze drier was used for the freeze-drying process. Freeze drying was started at 0.15 mbar after moringa flowers were placed on unheated shelves (15 Pa). The ambient radiation that reached the samples through the clear glass drying chamber provided the sublimation energy. The samples were then lyophilized for 24 hours at varied low temperatures, such as -40, -50, and -60°C. The freeze-dried powder was properly packed and stored at ambient temperature.

Statistical Analysis

Data analysis was performed in a completely randomized design (CRD) using SPSS 14.0 for Windows (SPSS, 2005).

RESULTS AND DISCUSSION

Proximate composition of fresh and freeze-dried moringa flower powder (FD- MFP).

The Proximate composition of moringa flowers included moisture - 74.95%, protein - 6.68g, crude fibre - 1.9 g, β - carotene - 18.85mg and ascorbic acid - 282.32 mg. The moringa flowers have much-valued nutraceutical properties, where the total antioxidant activity, total flavonoids and total phenols were 226.14mg 100 g^{-1}, 17.98mg RE g^{-1} extract and 63.19mg

GAE g^{-1} extract, respectively, and with mineral contents of calcium – 48.8, iron - 2.85, phosphorous - 92.18, potassium - 367.4 and magnesium 48.12 mg 100 g^{-1}.

Similar results were recorded by Lakshmipriya *et al.* (2016) through the chemical characteristics of the moringa flower which shows the protein 2.5 g, carbohydrate 3.7 g, fibre 4.8 g, vitamin C 120 mg, calcium 30 mg, magnesium 24 mg, phosphorus 110 mg, potassium 259 mg and iron 5.3 mg per 100 g. Shailendra *et al.*, 2016 analyzed the moringa and it contains moisture - 89.99 %, ascorbic acid - 127.39 mg per 100 g, calcium - 31.95 mg per 100 g, iron - 5.35 mg per 100 g, phosphorus - 107.85 mg per 100 g, crude fibre - 4.00 per cent and vitamin A - 185.06 IU and his results were accordance with the present investigation. Thurber and Fahey (2009) stated that *M. oleifera* parts are rich in protein, total carotenoids (40139 µg/100 g), nearly 47.8 % β-carotene, 6.6 mg/g ascorbic acid, 22.4 mg/g iron, 6.3 mg/g calcium and 0.9 g/100 g fibre which reduce malnutrition problems among the community and have great potential to combat micronutrient deficiencies.

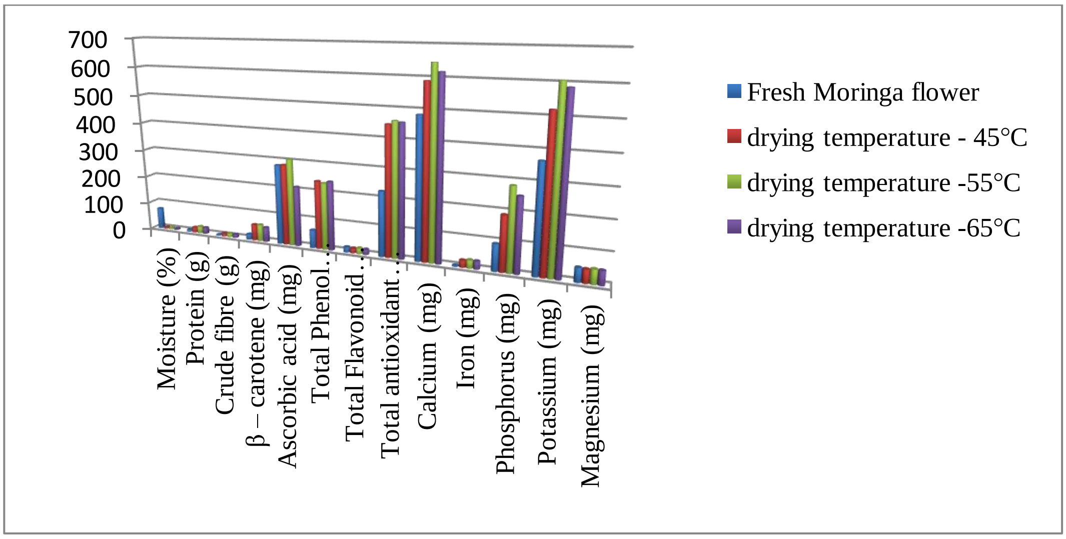
The Physico-chemical composition of the FD- MFP is presented in Table 1 and Figure 1**.** The moisture content of the MFP varied between 4.53 (45°C) and 4.075% (65°C). The protein and crude fibre contents of FD- MFP were 18.13 - 23.92and 9.88 -12.03 per 100 g, respectively, at different temperatures. The β – carotene content of powder dried at 55°C was higher (58.41 mg per 100 g) than those dried at 45°C and 65°C. Similarly, the ascorbic acid content was also higher (305.81mg per 100 g) in MFP dried at 55°C, followed by 45°C (285.09 mg) and 65°C(211.48 mg). The total phenol, flavonoid and antioxidant activities were 234.02 to 241.08 mg/GAE, 16.18 to 19.34 mg/RE and 448.12 to 460.18mg per 100 g, respectively, at different temperature levels. The maximum retention of calcium, iron, phosphorus, potassium and magnesium was found at drying temperatures of 45°C and 55°C compared with 65°C.

Table 1. Physicochemical characteristics of fresh and dried moringa flowers

ılars	**Fresh Ioringa flower**	**Different drying temperature**		
		- 45°C	**-55°C**	**-65°C**
re (%)	74.95	4.53	4.023	4.075
(g)	6.68	18.13	23.92	21.74
fibre (g)	1.9	9.88	12.03	10.89
otene (mg)	18.85	57.09	58.41	51.03
ic acid (mg)	282.32	285.09	305.81	211.48
henol content (mg/GAE)	63.19	239.02	234.13	241.08
lavonoid content (mg/RE)	17.98	16.18	19.34	18.11
ntioxidant activity (mg)	226.14	448.12	460.18	456.2
n (mg)	484.1	589.3	645.26	618.07
ıg)	2.85	23.75	27.5	26.63
orus (mg)	92.18	189.5	284.61	253.78
um (mg)	367.4	520.62	605.1	586.8
sium (mg)	48.12	46.17	49.58	48.21

The same results are in agreement with the literature reported. Manju *et al.,* 2021 in freeze-dried moringa flowers shows moisture - 3.70 ± 0.06, crude protein - 16.48 ± 0.34, crude fibre - 7.90b ± 0.16, total phenols - 192.21b ± 3.70 and vitamin C - 174.41d ± 2.89. Silva *et al.,* 2019 and Ademiluyi *et al.,* (2018) also reported the same results in freeze-dried moringa at different temperatures their results were Moisture content (5.88%), fat (8.15 %), crude protein (27.02%), crude fibre (13.39%) and carbohydrate (37.20 %) Potassium (2.40 %), Phosphorus (5.40 Mg/g), Iron (6.27 Mg/L), Magnesium (5.19 Mg/L) and Calcium (4.65%)

Figure 1. Physicochemical characteristics of fresh and dried moringa flowers



Raising the air-drying temperature (65°C) resulted in a greater loss of heat sensitivity nutrients such as carotene and ascorbic acids due to thermal deterioration and oxidation. Low moisture combined with a higher temperature for a longer period resulted in a greater reduction in TP and TAA. Increased phenolic content was associated with better antioxidant activity at optimal temperature due to the phenolic compound's high hydrogen atom-donating ability. When the drying temperature was raised above the optimal threshold, the leaf cell walls were damaged, causing the release of oxidative and hydrolytic enzymes, which destroyed the antioxidant properties.

Qualitative phytochemical screening of fresh moringa flower (Table 2),

The present qualitative screening results indicate phytochemicals were highly present in methanol and ethanol extracts, and a lower amount of phytochemicals was noticed in the chloroform extract. The same results were recorded by Bamishaiye *et al.,* (2011) and Nweze *et al.,* (2014) in methanol and ethanolic moringa extracts respectively. Intracellular ingredients are extracted from the cellular membrane by methanol and ethanol solvents due to their high penetration capacity (Wang *et al.,* 2009). In diluted ethanol, the concentration of flavonoids was higher due to amplified polarity (Aliyu *et al.*, 2016). Extraction of bioactive compounds from medicinal plants is limited to solvents (chloroform and hexane) with very low strength and polarity (Liu *et al.,* 2000). Among the phytochemicals, tannins and flavonoids were highly detected in methanol and ethanol extracts when compared to other solvent extracts such as chloroform, hexane, acetone and ethyl acetate. Okwu DE and Okwu ME 2004 stated the higher concentration of flavonoids in moringa parts will also help to protect against allergies, platelet aggregation, inflammation, free radicals, viruses, microbes, ulcers and tumours.

Table 2. Qualitative phytochemicals screening of fresh moringa flower

Phytochemicals	Different Extract					
	Methanol + Moringa Flower	Ethanol + Moringa Flower	Chloroform + Moringa Flower	Hexane + Moringa Flower	Acetone + Moringa Flower	Ethyl acetate + Moringa Flower
Alkaloids	+	+	-	+	-	+
Tannins	+++	+++	+	+	+	+
Flavonoids	++	+++	+	+	+	+
Steroids	++	+	+	+	+	-
Terpenoids	+	+	-	+	-	+
Saponin	+	+	-	+	+	+

Conclusion

For both fresh and dried moringa flower powder, the effects of drying temperature were investigated. The optimal drying temperature of 55°C results in improved physicochemical features such as – carotene (55.41 mg), total antioxidant (460.18 mg), and flavonoids (19.34 mg/RE), phenol (234.13 mg/GAE), and vitamin C content (485.09 mg). According to the findings of this study, FD drying is one of the best drying techniques for preserving nutritional quality features while also being efficient and cost-effective for the entire food processing industry. Moringa flower powder, which has been freeze dried, can be used to make novel functional foods including vegetable soup, energy bars, energy drinks, extruded goods, and incorporated food products that can be employed in nutritional intervention programmes.

References

Abbas, R. K., Elsharbasy, F. S. and Fadlelmula, A. A. Nutritional Values of Moringa oleifera, Total Protein. Amino Acid, Vitamins, Minerals, Carbohydrates, Total Fat and Crude Fiber, under the Semi-Arid Conditions of Sudan. *J. Microb. Biochem. Technol.*, **(2018),** *10*, 56-58. DOI: 10.4172/1948-5948.1000396.

Ademiluyi, A. O., Aladeselu, O. H., Oboh, G. and Boligon, A. A. Drying alters the phenolic constituents, antioxidant properties, α-amylase, and α-glucosidase inhibitory properties of Moringa (Moringa oleifera) leaf. *Food science & nutrition*, **(2018).** 6(8), 2123-2133.

Aliyu, A., Chukwuna, U. D., Omoregie, E. H. and Folashade, K. O. Qualitative phytochemical analysis of the leaf of Moringa oleifera lam. from three climatic zones of Nigeria. *Journal of Chemical and Pharmaceutical Research*, **(2016).** *8*(8), 93-101.

Andzouana, M. and Mombouli, J. B. Assessment of the chemical and phytochemical constituents of the leaves of a wild vegetable-Ochthocharis dicellandroides (Gilg). *Pakistan Journal of Nutrition*, **(2012).** *11*(1), 94-99. http://pjbs.org/pjnonline/fin2202.pdf.

Anwar F, Latif S, Ashraf M, Gilani AH. *Moringa oleifera:* a food plant with multiple medicinal uses. *Phytotherapy Res,* **(2007)**, 21:17-25.

AOAC. **(1990).** Official Methods of Analysis. Association of Official Analytical Chemists, Washington D.C.

Arun Prabhu, R., Anand Prem Rajan and Sarita Santhalia. Comparative analysis of preservation techniques on *Moringa oleifera*. *Asian Journal of Food and Agro-Industry*, **(2011),** 4(02), 65-80.

Azevedo, Í. M., Araújo-Filho, I., Teixeira, M. M. A., Moreira, M. D. F. D. C. and Medeiros, A. C. Wound healing of diabetic rats treated with *Moringa oleifera* extract. *Acta cirúrgica brasileira*, **(2018).** *33*, 799-805.

Bamishaiye E. I., Olayemi F. F., Awagu E. F. and Bamishaiye O. M. Proximate and phytochemical composition of *Moringa oleifera* leaves at three stages of maturation. *Adv. J. Food Sci. Technol*, **(2011).** 3(4), 233- 237.

Barimah, J., Yanney, P., Laryea, D. and Quarcoo, C.. Effect of drying methods on phytochemicals, antioxidant activity and total phenolic content of dandelion leaves. *J Food Nutr*, **(2017)** 5(4), 136-141.

Boudieb, Kaissa, Sabrina Ait Slimane-Ait Kaki. And Hayet Amellal-Chibane. "Effect of Maturation Degree on the Fixed Oil Chemical Composition, Phenolic Compounds, Mineral Nutrients and Antioxidant Properties of Pistacia lentiscus L. Fruits." *Notulae Botanicae Horti Agrobotanici Cluj-Napoca*, **(2019).** 47(3), 836-847. https://doi.org/10.15835/nbha47311534

Chen, J., Li, F., Li, Z., McClements, D. J. and Xiao, H. Encapsulation of carotenoids in emulsion-based delivery systems: Enhancement of β-carotene water-dispersibility and chemical stability. *Food Hydrocolloids*, **(2017).** *69*, 49-55. https://doi.org/10.1016/j.foodhyd.2017.01.024

Kinki, A., Mezgebe, A. and Lema, T. Antioxidant and Sensory Properties of Herbal Teas Formulated from Dried Moringa (MoringaStenopetala) and Stevia (Stevia Rebaudiana Bertoni) Leaves. **(2020),** 10.7176/FSQM/102-01.

Klungboonkrong, V., Phoungchandang, S. and Lamsal, B. Drying of Orthosiphonaristatus leaves: Mathematical modeling, drying characteristics, and quality aspects. *Chemical Engineering Communications*, **(2018),** 205(9), 1239-1251. https://doi.org/10.1080/00986445.2018.1443080

Lakshmipriya Gopalakrishnan, Kruthi Doriya and Devarai Santhosh Kumar. *Moringa Oleifera*: A review on nutritive importance and its medicinal application. *Food Science and Human Wellness*, **(2016).** 5(2), 49-56. https://doi.org/10.1016/j.fshw.2016.04.001

Lim Y. Y., T. T. Lim and J. J. Tee. Antioxidant properties of several tropical fruits: A comparative study. *Food Chemistry*, **(2007).** 103, 1003-1008. https://doi.org/10.1016/j.foodchem.2006.08.038

Liu, F. F., Ang, C. Y. and Springer, D. Optimization of extraction conditions for active components in Hypericum perforatum using response surface methodology. *Journal of agricultural and food chemistry*, **(2000).** 48(8), 3364-3371. https://doi.org/10.1021/jf991086m

Manju, K. M., and Kumar, N. Effect of fluidized-bed and freeze-drying techniques on physicochemical, nutritional, thermal, and structural properties of *Moringa oleifera* flowers, leaves, and seeds. *Journal of Food Processing and Preservation*, **(2021)** 45(9), e15719.

Marcel, A., Hubert, M., Bienvenu, M. J. and Pascal, O. Physicochemical characteristics and biochemical potential of *Moringa oleifera* Lam.(Moringaceae). *Der Pharmacia Lettre*, **(2016).** 8(18), 43-47. https://doi.org/10.1111/jfpp.13915

Meda, A., Lamien, C.E., Romito, M., Millogo, J. and O. G. Nacoulma, Determination of the total phenolic, flavonoid and proline contents in Burkina Fasan honey, as well as their radical scavenging activity. *Food Chemistry*, **(2005).** 91(3), 571–577. https://doi.org/10.1016/j.foodchem.2004.10.006

Nweze, N. O. and Nwafor, F. I. Phytochemical, proximate and mineral composition of leaf extracts of *Moringa oleifera* Lam. from Nsukka, South-Eastern Nigeria. **(2014).**

Okwu D. E. and Okwu M. E. Chemical composition of *Spondias mombin* Linn plant parts. *J. Sustain. Agric. Environ*, **(2004).** 6, 140-147.

Quettier - Deleu C., Gressier, B., Vasseur, J., Dine, T., Brunet, C. and Luyckx MPhenolic compounds and antioxidant activities of buckwheat (*Fagopyrum sculentum* Moench) hulls and flour. *Journal of Ethnopharmacology*, **(2000).** 72, 35-42. https://doi.org/10.1016/S0378-8741(00)00196-3

Ranganna S. Manual of analysis of fruits and vegetables products. Tata McGraw Hill publishing Co, Ltd., New Delhi. **(1995).** P. 1-2, 7- 13.

Sekhar, C., Venkatesan, N., Murugananthi, D. and Vidhyavathi, A. Status of Value Addition and Export of Moringa Produce in Tamil Nadu A Case Study. *Economic Botany,* **(2018),** 34(3), 276-283.

Shailendra Patni, Krishan Kumar Bijarnia, Harsh Enaniya, Kailash Sharma, Bhanwar Lal and Raaz K Maheshwari. Drumstick tree: A miracle forwell-beingand sociopecuniary diverse therapeutic applicability. *International Journal of Chemistry and Pharmaceutical Sciences,* **(2016).** 4(2), 108 - 114.

Silva, M. A., Ayed, C., Foster, T., Camacho, M. D. M., and Martínez-Navarrete, N. The impact of freeze-drying conditions on the physico-chemical properties and bioactive compounds of a freeze-dried orange puree. *Foods*, **(2019).** 9(1), 32.

Thurber, M. D. and J. W. Fahey. Adoption of *Moringa oleifera* to combat undernutrition viewed through the lens of the "Diffusion of Innovations" theory. *Ecol. Food Nutr,* **(2009).** 48, 212-225. https://doi.org/10.1080/03670240902794598

Wang, G. X., Han, J., Feng, T. T., Li, F. Y. & Zhu, B. Bioassay-guided isolation and identification of active compounds from Fructus Arctii against Dactylogyrus intermedius (Monogenea) in goldfish (*Carassius auratus*). *Parasitology research*, **(2009).** *106*(1), 247-255. https://doi.org/10.1007/s00436-009-1659-7