



P-ISSN: 2349-8528

E-ISSN: 2321-4902

[www.chemijournal.com](http://www.chemijournal.com)

IJCS 2021; 9(1): 1455-1459

© 2021 IJCS

Received: 12-10-2020

Accepted: 23-11-2020

**Nilesh Dehariya**Department of Agricultural and  
Food Engineering, Indian  
Institute of Technology  
Kharagpur, West Bengal, India**Proshanta Guha**Department of Agricultural and  
Food Engineering, Indian  
Institute of Technology  
Kharagpur, West Bengal, India**Rakesh Kumar Gupta**Department of Agricultural and  
Food Engineering, Indian  
Institute of Technology  
Kharagpur, West Bengal, India

## Extraction and characterization of essential oil of garlic (*Allium sativa* L.)

Nilesh Dehariya, Proshanta Guha and Rakesh Kumar Gupta

DOI: <https://doi.org/10.22271/chemi.2021.v9.i1u.11426>

### Abstract

The essential oil was extracted from garlic powder by soxhlet extraction method using ethanol as a solvent. The yield of essential oil was influenced by extraction time and temperature. The maximum extraction yield was 16.55% under treatment- T7 (50 °C for 4 hours). The density and refractive index of the oil was 0.875 (g/ml) and 1.52 respectively. The oil is light yellowish in colour with a pungent odour. The maximum TOAC (12.01 mM  $\alpha$  tocopherol per ml of essential oil) was found under T3 treatment. Essential oil was analyzed by gas chromatography–mass Spectrometry (GC-MS). The major chemical compound were: diallyl disulfide (48.42%), allyl methyl trisulfide (7.27%), trisulfide, di-2 propenyl (3.46%), and diallyl sulfide (7.64%). The results revealed that T7 was the best treatment among all treatments. This indicates the feasibility of garlic oil production at a commercial scale for culinary and medical utilization.

**Keywords:** Garlic essential oil, chemical composition, refractive index, TOAC, *Allium sativum*

### 1. Introduction

*Allium sativum* L., commonly known as garlic, belongs to the onion family, *liliaceae*. Garlic was likely originated in Central Asia and it has been in use throughout the world for both culinary and medicinal purposes [1, 2]. The garlic oil, rich in sulfureted organic compounds, contains a variety of sulfides such as diallyl disulfide and diallyl trisulfide [3-7]. It is used not only as a flavoring agent, food preservative but also in the prevention and treatment of several illnesses [8, 9]. In the pharmaceutical industry, it is much used due to its anticarcinogenic, antithrombotic and antiplatelet aggregation properties. The regular consumption of garlic oil can reduce blood pressure, prevent heart disease including atherosclerosis, high cholesterol and cancer [10]. Recent biological and pharmacological research [11-21] confirms these medicinal properties showing that garlic oil has an antibiotic, antioxidant, anti-viral, anti-fungal, antimicrobial, anticarcinogenic and immunomodulatory effect and garlic can be used to prevent nausea, diarrhea, ease coughs and even in treatment in conditions such as malaria and cholera. It is an immune system enhancer [22]. Some studies have found lower rates of certain types of cancer in people who use it regularly.

Being the second largest producer of garlic, India maintains surplus quantify most of the times that remains unutilized. India provides 5.2% of the total world production followed by China with 80% share in the global market. The other major producers are Bangladesh and Egypt followed by Korea, Russia and others. In India Madhya Pradesh, Rajasthan, Gujarat, Orissa, Uttar Pradesh and Maharashtra are the main states where garlic is grown commercially with an average yield of 6-8 tonnes/ha. Madhya Pradesh is the leading garlic producing state with the production of 4.24 lakh tones accounting to about 26.25% of total Indian production and a yield of 7.86 tonnes/ha.

In India, due to lack of poor post-harvest handling practices, suitable storage, processing facilities, heavy losses are incurred both in terms of quality and quantity. This may be attributed to respiration, transpiration and microbiological spoilage. Though garlic is produced abundantly and consumed as such, little efforts have so far been made to produce garlic essential oil from garlic powder. Garlic is a semi perishable commodity and nearly 30% of the crop is wasted due to respiration and microbiological spoilage during storage [23], which needs to be addressed. Therefore, it is important to diversify its utility forms.

**Corresponding Author:****Nilesh Dehariya**Department of Agricultural and  
Food Engineering, Indian  
Institute of Technology  
Kharagpur, West Bengal, India

Extraction of essential oil is a major food processing operation in the food industry for utilization of this surplus garlic in terms of value addition and income generation and thereby minimize wastage.

There are different methods for extraction of essential oil. In this study, the essential oil was extracted by soxhlet extraction method using ethanol as a solvent. This study had four objectives: 1) Optimization of a process parameter, 2) Determination of physical properties, 3) To find out chemical composition of the extracted oil, 4) Evaluation of antioxidant capacity.

## 2. Materials and Methods

Fresh garlic (local variety) was procured from Tech market, IIT Kharagpur and during the experiments, all the samples were stored in our lab at appropriate conditions (dark, 27 °C). The garlic cloves were cut into two equal-size manually by stainless steel knife with the utmost care and immediately kept into the oven at 60 °C to dry for 48 hours and the powder was made by mechanical tools.

### 2.1. Extraction of Essential Oil

The solvent extraction method was conducted with a soxhlet extractor using commercial ethanol at different temperature of 50, 60 and 70 °C for 2, 3 and 4 hours. The combination of temperature and time were determined in a preliminary set of experiments. Three replicates were carried out for all nine treatments to reduce the error. Garlic powder was used (10g) at 1:20, sample to solvent ratio. The oil was obtained after the solvent was evaporated by placing over a water bath (LABARD, LI-WBPR-14A) for about 2-3 hours under reduced temperature (50 °C) and refluxing at 70 °C to remove any excess solvent [2]. The extracted garlic oil was stored in a refrigerator at 4 °C for subsequent physico-chemical analyses.

### 2.2. Antioxidant Activity

The total antioxidant capacity (TAOC) of the essential oil samples was measured as previously described [24]. Briefly, 40 µl of essential oil was mixed with a reagent solution (0.6 N sulphuric acid, 28 mM sodium phosphate, and 4 mM ammonium molybdate) in an Eppendorf tube in the ratio of 1:100 (v/v) and the tubes were capped and incubated at 95 °C for 90 minutes. The addition of essential oil to the reagent solution caused discoloration after incubation. This indicates the scavenging capacity of essential oil. Samples were cooled at room temperature and absorbance was measured at 695 nm using a spectrophotometer (Epoch 2, BioTek, U.S.A.). All determinations were performed in duplicate. TAOC of the samples were expressed as equivalents of mM- $\alpha$ -tocopherol per ml of essential oil and was calculated as follows:

$$A = \epsilon CL$$

Where

A = Absorbance

$\epsilon$  = Extinction co-efficient (4 × 10<sup>3</sup> M<sup>-1</sup> cm<sup>-1</sup>)

C = Concentration (molar)

L = Path length (1 cm)

$$TOAC = \frac{A/(\epsilon \times L)}{\text{Amount of sample}} \text{ mM}/\alpha \text{ tocopherol/ml}$$

### 2.3. Chemical Composition

The volatile oil extracted from garlic powder was subjected to GC-MS analysis as previously described [25-32]. A GC-MS

(Thermo Scientific, Trace 1300), GC (TRACE-GC ULTRA) and MS (POLARISQ) instrument was used to study the composition of extracted essential oil. This instrument was operated in the electron impact (EI) mode set at electron energy 70 eV and a scan range of 0.00 amu–100 amu, with a scan rate of 3.0 scans per second. DB-5MS column of 30 m length with 0.25 mm inner diameter was used. Helium gas (99.99%) was used as a carrier at a constant flow rate of 1 ml min<sup>-1</sup> on the column head. The temperature of the injector was set at 250 °C and the temperature of the ion source was set at 230 °C. The temperature of the GC oven was programmed to be 50 °C initially and was programmed to increase at a rate of 5 °C/min to a final temperature of 260 °C. The sample was prepared by diluting the essential oil in a ratio of 1:10 with methanol and 1.0 µl volume of sample was injected into the instrument with a split ratio of 30:1.

The obtained mass spectra were thoroughly screened and individual components of essential oils were quantified by relative peak percent area. Identification of each quantified component was done by comparing their mass fragmentation pattern with components stored in the spectrometer database using NIST mass spectral library (Version 2014).

### 2.4 Removal of Milky Emulsion and Excess Solvent

Since the milled substrate showed a tendency to agglomerate during extraction, optimal particle size was determined in a preliminary set of experiments as the smallest that did not cause perceptible agglomeration problems and this size was 1-3 mm. Some essential oil extracted with milky emulsion (Fig. 1) was centrifuged at 3000 rpm for 5 minutes by a high-speed refrigerated research centrifuge –RC 4100 F. By this, the milky emulsion stuck around the surface of the bottle and clean essential oil including solvent was separated by pipet, then placed in a water bath for removing excess solvent.



Fig 1: Essential oil + Solvent+ Emulsion

## 3. Results and Discussion

### 3.1. Yield of Garlic Oil

The yield of garlic essential oil changed with the process temperature, and time (Table 1). The best treatment for this research work was T7 (50 °C for 4 hour). The maximum extraction yield was 16.55% (db) among the nine treatments. By optimizing the process parameter, the best combination of the parameters were found to be 4 hours and 50 °C. These parameters can be ideal one to obtain the maximum yield at a commercial level. Thus, in the present study much higher oil yield was obtained compare to that reported by Ali Rafe *et al.* 2014 [33]. They found that maximum yield were 5.5, 6 and 7% for steam distillation, solvent method and SCF-CO<sub>2</sub>, respectively.

**Table 1:** Extraction yield %

Treatment	Time (h)	Temperature (°C)	Yield (%)
T1	2	50	7.55
T2	2	60	9.33
T3	2	70	11.88
T4	3	50	14.55
T5	3	60	13.55
T6	3	70	13.75
T7*	4	50	16.55
T8	4	60	15.88
T9	4	70	15.33

### 3.2. Physical properties

The Physical examination of the extracted oil was conducted and presented in Table 2. The properties were moisture content, density, refractive index, appearance and odour. The moisture content of the peeled garlic cloves was (63.14±1%). The density and refractive index were 0.875±1 (g/ml) and 1.52 at room temperature which falls within the range of volatile oil in general. The appearance of extracted oil was light yellowish and it had pungent odour.

**Table 2:** Physical properties of extracted garlic essential oil

Parameters	Results
Moisture content (%)	63.14±0.38
Density (g/cm <sup>3</sup> )	0.873±0.003
Refractive index	1.52±0.02
Appearance	Light yellow
Odour	Pungent
Oil yield (%)	16.55±0.33

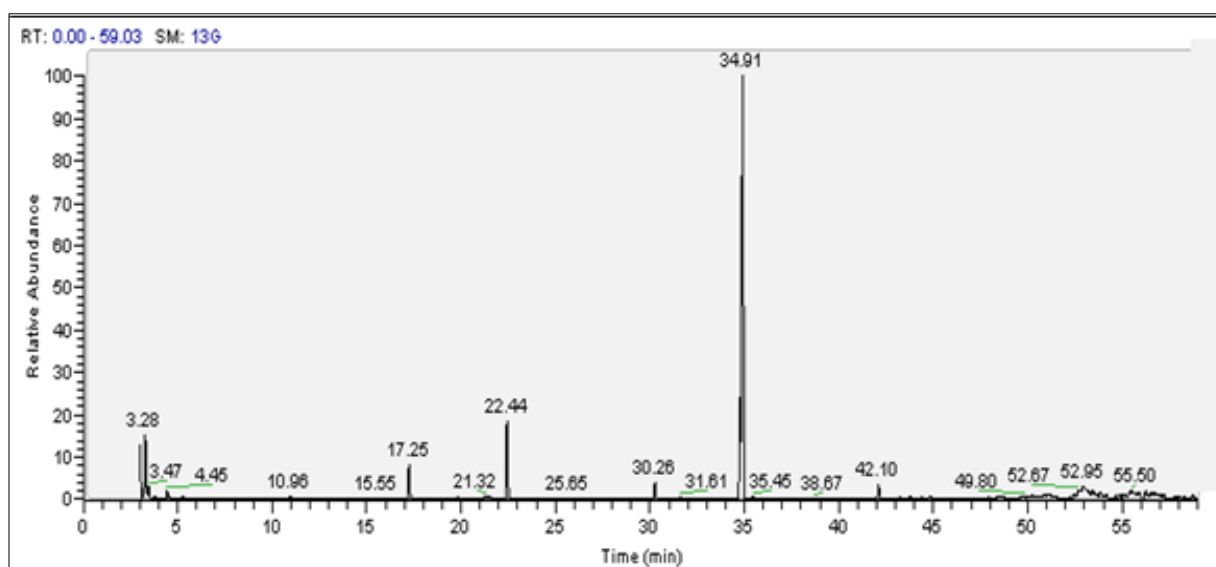
### 3.3. Chemical Composition of Extracted Oil

The essential oil compositions were determined by GC-MS. The qualitative and quantitative differences of compounds are

presented in Table 3. The total ion chromatogram of garlic essential oil is shown in Fig. 3. The major compounds in the extracted oil were diallyl disulfide (48.42%), allyl methyl trisulfide (7.27%), trisulfide, di-2 propenyl (3.46%) and daillyl sulfide (7.64%). There was some loss of volatile compounds of essential oil, may be during oven drying of garlic slices. Indeed these chemical compounds covered more than 85% of GC profile. Sulfide compound was dominated among all compounds. This result is slightly different from other authors (Douiri *et al.* 2013) [34] who reported that garlic essential oil obtained by Clevenger hydrodistillation contained diallyl disulfide (16.0%) and allyl methyl trisulfide (10.9%). Similarly, Rao *et al.* 2007 [35] have analyzed six geographical varieties of essential oils extracted by steam distillation from fresh garlic grown in India and found that diallyl disulfide (27.1–46.8%) and diallyl trisulfide (19.9–34.1%) dominated in the oil followed by allyl methyl trisulfide (8.3–18.2%) and allyl methyl disulfide (4.4–12.0%). It can be expected that this oil may be commercialized for medicinal purposes in view of its reported prophylactic and curative profile.

**Table 3:** Results of GC-MS analyses of extracted oil

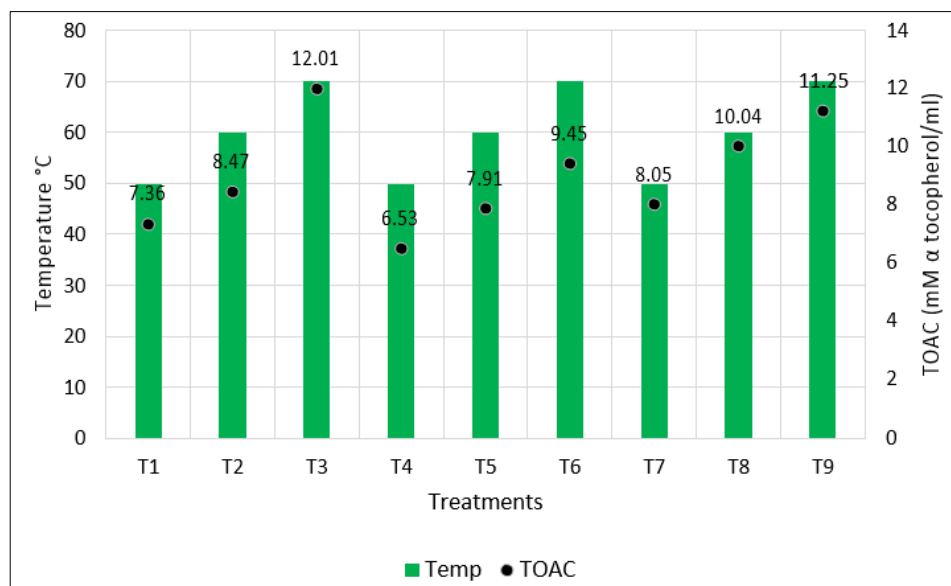
RT (min)	Compounds	Composition %
34.91	Diallyl disulfide,	48.42%
3.28	Daillyl sulfide	7.64%
22.44	Ally methyl trisulfide	7.27%
17.25	Trisulfide, di-2 propenyl	3.46%
55.94	Hydrazine, methyl	5.75%
56.24	2-Propanone, 1-hydroxy	5.81%
51.10	1, 2-Cyclopentanedione	1.24%
53.43	Cyclopropane carboxylic acid 1-amino	0.52%
52.95	Benzoic acid, 2-methyl	3.22%
58.08	3-Vinyl-1, 2-dithiacyclohex-5-en	1.13%
54.17	1H-Pyrrole, 1-methyl	0.75%

**Fig 2:** Total ion chromatogram of the garlic essential oil (peak assignments are given in table)

### 3.4. Antioxidant capacity

The total antioxidant capacity of essential oil was analyzed for each treatment and presented in Fig. 2. The maximum total antioxidant capacity was 12.018 mM  $\alpha$  tocopherol per ml of essential oil for T3 treatment (70 °C for 2 hours). The results indicate that for a specific duration of time with every

10 °C rises in temperature there was an increase in antioxidant activity. It may be due to phenolic compound and the sulfur compound was more active at 70 °C temperature as compare to 50 °C and 60 °C temperature or these compound may be extracted more at 70 °C.



**Fig 3:** Effect of temperature and time on total antioxidant capacity

#### 4. Conclusions

This study has demonstrated that there is a significant impact of temperature and time on the yield of essential oil. The best parameters are 4 hours and 50°C for obtaining the maximum yield. These parameters can be considered as an ideal one for commercial production. The physical properties i.e. moisture content, density, refractive index, appearance and odour were within the range of essential oil. The refractive index of extracted oil can be used for its identification from other edible oil sources. These properties indicates the feasibility of garlic oil production for commercial purposes. The extracted oil was found to be a very good antioxidant and it can not only be used for food preservation but also for prophylactic and therapeutic uses. The principal chemical compounds detected were: diallyl disulfide (48.42%), allyl methyl trisulfide (7.27%), trisulfide, di-2 propenyl (3.46%), and diallyl sulfide (7.64%). Sulfide compound dominated among the all compounds. It can be expected that this oil can be commercialized for medicinal and culinary purposes.

#### 5. Acknowledgements

The authors are grateful to the Indian Institute of Technology Kharagpur for financial assistance, infrastructure and facilities to conduct the research. They are also thanks to Dr. D. K. Swain, Prof. S. L. Srivastava, Mr. Sujosh Nandi, Mr. Jagan Kartik S, Ms. Mitali Madhumita and Ms. Kalyani Hemraj Burde, of Agricultural and Food Engineering Department, IIT Kharagpur for their continued support.

**6. COI Statement:** The authors declared that they have no conflict of interest

#### 7. References

- McGee H. On food and cooking: the science and lore of the kitchen. Simon and Schuster 2007.
- Gafar MK, Itodo AU, Warra AA, Abdullahi L. Extraction and physicochemical determination of garlic (*Allium sativum* L) oil. Int J Food Sci Nutr 2012;1(2):4-7.
- Huang Y, Chen SX, Ho SH. Bioactivities of methyl allyl disulfide and diallyl trisulfide from essential oil of garlic to two species of stored-product pests, *Sitophilus zeamais* (Coleoptera: Curculionidae) and *Tribolium castaneum* (Coleoptera: Tenebrionidae). Journal of Economic Entomology 2000;93(2):537-543.
- Jo KS, Kim HK, Ha JH, Park MH, Shin HS. Flavor compounds and storage stability of essential oil from garlic distillation. Korean Journal of Food Science and Technology 1990;22(7):840-845.
- Kim JW, Kim YS, Kyung KH. Inhibitory activity of essential oils of garlic and onion against bacteria and yeasts. Journal of food protection 2004;67(3):499-504.
- Satyap P, Craft JD, Dosoky NS, Setzer WN. The chemical compositions of the volatile oils of garlic (*Allium sativum*) and wild garlic (*Allium vineale*). Foods 2017;6(8):63.
- Lawrence R, Lawrence K. Antioxidant activity of garlic essential oil (*Allium sativum*) grown in north Indian plains. Asian Pacific Journal of Tropical Biomedicine 2011;(z1):51-54.
- Seydim AC, Sarikus G. Antimicrobial activity of whey protein based edible films incorporated with oregano, rosemary and garlic essential oils. Food research international 2006;39(5):639-644.
- Del Valle JM, Mena C, Budinich M. Extraction of garlic with supercritical CO<sub>2</sub> and conventional organic solvents. Brazilian Journal of Chemical Engineering 2008;25(3):532-542.
- Avato P, Tursi F, Vitali C, Miccolis V, Candido V. Allylsulfide constituents of garlic volatile oil as antimicrobial agents. Phytomedicine 2000;7(3):239-243.
- Benkeblia N. Antimicrobial activity of essential oil extracts of various onions (*Allium cepa*) and garlic (*Allium sativum*). LWT-food science and technology 2004;37(2):263-268.
- Cavallito CJ, Bailey JH, Buck JS. The antibacterial principle of *Allium sativum*. III. Its precursor and essential oil of garlic. Journal of the American Chemical Society 1945;67(6):1032-1033.
- Casella S, Leonardi M, Melai B, Fratini F, Pistelli L. The role of diallyl sulfides and dipropyl sulfides in the *in vitro* antimicrobial activity of the essential oil of garlic, *Allium sativum* L., and leek, *Allium porrum* L. Phytotherapy Research 2013;27(3):380-383.
- Corzo-Martínez M, Corzo N, Villamiel M. Biological properties of onions and garlic. Trends in food science & technology 2007;18(12):609-625.
- Dziri S, Casabianca H, Hanchi B, Hosni K. Composition of garlic essential oil (*Allium sativum* L.) as influenced

- by drying method. *Journal of Essential Oil Research* 2014;26(2):91-96.
16. Edris AE, Fadel HM. Investigation of the volatile aroma components of garlic leaves essential oil. Possibility of utilization to enrich garlic bulb oil. *European Food Research and Technology* 2002;214(2):105-107.
  17. Kim JW, Huh JE, Kyung SH, Kyung KH. Antimicrobial activity of alk(en)yl sulfides found in essential oils of garlic and onion. *Food Science and Biotechnology* 2004;13(2):235-239.
  18. Moreno FJ, Corzo-Martí M, Del Castillo MD, Villamiel M. Changes in antioxidant activity of dehydrated onion and garlic during storage. *Food Research International* 2006;39(8):891-897.
  19. Romeilah RM, Fayed SA, Mahmoud GI. Chemical compositions, antiviral and antioxidant activities of seven essential oils. *J Appl Sci Res* 2010;6(1):50-62.
  20. Bozin B, Mimica-Dukic N, Samojlik I, Goran A, Igc R. Phenolics as antioxidants in garlic (*Allium sativum* L., Alliaceae). *Food chemistry* 2008;111(4):925-929.
  21. Sharma GP, Prasad S, Chahar VK. Moisture transport in garlic cloves undergoing microwave-convective drying. *Food and bioproducts processing* 2009;87(1):11-16.
  22. Bagudo BU, Acheme OD. Chemical analysis of locally cultivated garlic and its oil. *Der Chemica Sinica* 2014;5(1):128-134.
  23. Prieto P, Pineda M, Aguilar M. Spectrophotometric quantitation of antioxidant capacity through the formation of a phosphomolybdenum complex: specific application to the determination of vitamin E. *Analytical biochemistry* 1999;269(2):337-341.
  24. Arabshahi-Delouee S, Urooj A. Antioxidant properties of various solvent extracts of mulberry (*Morus indica* L.) leaves. *Food chemistry* 2007;102(4):1233-1240.
  25. Andreatta AE, Foco G, Mabe G, Bottini SB. Extraction of garlic oil with Quasi-Critical Solvents. *Enproma, Argentina* 2005, 1-9.
  26. Avato P, Miccolis V, Tursi F. Agronomic evaluation and essential oil content of garlic (*Allium sativum* L.) ecotypes grown in Southern Italy. *Advances in horticultural science* 1998, 201-204.
  27. Calvo-Gómez O, Morales-López J, López MG. Solid-phase microextraction– gas chromatographic-mass spectrometric analysis of garlic oil obtained by hydrodistillation. *Journal of Chromatography A* 2004;1036(1):91-93.
  28. Jirovetz L, Jäger W, Koch HP, Remberg G. Investigations of volatile constituents of the essential oil of Egyptian garlic (*Allium sativum* L.) by means of GC-MS and GC-FTIR. *Zeitschrift für Lebensmittel-Untersuchung und Forschung* 1992;194(4):363-365.
  29. Kimbaris AC, Siatis NG, Daferera DJ, Tarantilis PA, Pappas CS, Polissiou MG. Comparison of distillation and ultrasound-assisted extraction methods for the isolation of sensitive aroma compounds from garlic (*Allium sativum*). *Ultrasonics sonochemistry* 2006;13(1):54-60.
  30. Grewal JS, Alam MS, Ubhi GS. Optimization of Process Parameters for Convective-cum-microwave Dehydration of Garlic Slices (*Allium sativum* L.). *International Journal of Engineering Science Invention* 2014;3:46-55.
  31. Li R, Chen WC, Wang WP, Tian WY, Zhang XG. Extraction of essential oils from garlic (*Allium sativum*) using ligarine as solvent and its immunity activity in gastric cancer rat. *Medicinal Chemistry Research* 2010;19(9):1092-1105.
  32. Sharma GP, Prasad S. Optimization of process parameters for microwave drying of garlic cloves. *Journal of Food Engineering* 2006;75(4):441-446.
  33. Rafe A, Nadjafi MS. Physicochemical characteristics of garlic (*Allium sativum* L.) oil: Effect of extraction procedure. *International Journal of Nutrition and Food Sciences* 2014;(3):6-1.
  34. Douiri L, Boughdad A, Assobhei O, Moumni M. Chemical composition and biological activity of *Allium sativum* essential oils against *Callosobruchus maculatus*. *IOSR J Environ. Sci. Toxicol. Food Technol* 2013;3:30-36.
  35. Rao PP, Nagender A, Rao LJ, Rao DG. Studies on the effects of microwave drying and cabinet tray drying on the chemical composition of volatile oils of garlic powders. *European Food Research and Technology* 2007;224(6):791.