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Original Paper

Heat-reflux processing of black peppercorn into bioactive antioxidant oleoresins: a three-functioned Taguchi-based grey relational grading

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Abstract

The focus of this research is to identify the best set of factors that influence the heat-reflux recovery of total phenolic content and antioxidant activities under multiple quality characteristics. **AQ1** Parametric Taguchi L_9 orthogonal design and grey relational analysis technique were used to investigate the effect of three variables—reflux duration, particle size, and feed-to-solvent ratio on the multiple responses of total phenolic contents, DPPH, and H_2O_2 activities. According to the grey relational grades response table, the ideal number of criteria for the heat reflux results were 120 min of reflux duration, 0.2 mm of particle size, and a feed-solvent ratio of 1:16. The total phenolic content, DPPH, and H_2O_2 scavenging activities were measured as 35.23 ± 0.004 mgGAE/g d.w, 107.57 ± 0.04 g/mL, and 87.78 ± 0.32 g/mL, respectively. Moreover, with the Levenberg–Marquardt (LM) neural network architecture, the trained network has a mean square error (MSE) of 3.7646E−07 and an R^2 of 0.9500 as the training function outcome, indicating a significant predicted endpoint. The confirmatory experimental results show a 41.9 per cent improvement in relation to the predicted values. The results of this study indicated that, optimising the heat reflux process would be an innovative and beneficial approach for preparing bioactive compounds from functional plants, resulting in cost **AQ2** savings while increasing antioxidant capacity and overall phenolic recovery.

Keywords

Antioxidants
Black pepper
Grey relational analysis (GRA)
Artificial neural network
Optimization
Total phenolic content

Introduction

Black peppercorns are powerful, hot-tasting, concentrated spice that is usually dried and powdered and is used to spice meats, seafood, veggies, condiments, stews, marinade, noodles, and other foods. Due to their nutraceutical potential, its by-products have been reported useful as food additives in form of oleoresin paste with many numerous health and nutritional **AQ3** benefits [1]. The growing demand for pepper offers a remarkable business opportunity for new marketers to enter the industry. Owning to this marginal interest and surge in the market value of black pepper there is an urgent need for its nutraceutical's diversification into other value-added by-products. One of such product diversifications is the extraction of spice oleoresins from black pepper consisting of both resin and essential oils with high potential use as feedstocks in food and pharmaceutical industries. Black pepper contains carbohydrates (37.4%), proteins (25.5%), fibre (23.6%), moisture (4.7%), and fat (5.3%), in addition to minerals such as potassium (0.66%), calcium (0.20%), phosphorus (0.16%), and magnesium (0.16%). Terpenes are the most significant volatile flavour and aromatic components in black pepper containing nitrogen-containing limonene, linalool, methyl-propanal, butyric acid, and 3-methylbutyric acid. Moreover, bioactive compounds such as 2,3-diethyl-5-methylpyrazine and 2-isopropyl-3-methoxypyrazine are the main component for black pepper's pungency and flavoury capacity.

Furthermore, the presence of antioxidants in the oleoresin extracts (fixed oil) offered a remarkable advancement for the treatment of some life-threatening free radical disorder such as cancer, diabetes, cardiovascular and neurological diseases [1]. The inherent antioxidant bioactive compounds in black pepper have also been reported to prevent chemical carcinogenesis via activation of xenobiotic enzymes [2]. Hence the dietary intake of black pepper extracts could **AQ4** provide natural antioxidants for boosting human immunity and prevent degenerative diseases [3]. The substantial amount of antioxidants in black pepper is largely associated with their total phenolic contents as reported by Poompavai and Gowri [4].

To adequately harness these functional properties, the hydro-distillation techniques has been previously used for recovering natural antioxidants from tough nuts, wood, seeds and hard surface powdered natural products [5]. The demerit from hydro-distillation method has provoked recent research focus into its heat-refluxing re-configuration. Take for instance, spice oils were extracted from white pepper and black pepper and tested for antioxidant activity by Abd El Mageed et al. [6]. The antioxidant properties of the spice oil were shown to diminished throughout the hydro distillation process. However, Khajeh and Ghanbari [7] reported that the difficulties in heat control are the major drawback in the use of hydro distillation which could result in extraction rate variation and hence a low extract quality. In heat reflux, the liquid is boiled, but the vapour is allowed to condense and flow back into the original flask. Allowing the condensed vapour to flow back into the original reaction flask, helps in the solvation of high molecular compounds. The continual recycling of the solvent helps reduce overheating by providing a cooling effects and drive the extraction to completion [8].

Furthermore, many researchers had studied the effect of extraction factors on the total phenolic content and antioxidants in black peppercorns. However, there has been little effort on the quality interaction between them at the response setting level [6]. It's also important to know that there is a correlation between the total phenolic and antioxidant activity with its combined effects determining its functional and therapeutic properties [9]. Previous studies conducted on the black pepper only investigated the antioxidant and total