www.ThePharmaJournal.com
The Pharma Innovation

ISSN (E): 2277- 7695
ISSN (P): 2349-8242
NAAS Rating: 5.03
TPI 2021; 10(2): 256-261

www.thepharmajournal.com
Received: 07-12-2020
Accepted: 15-01-2021

Priti Jain
Department of Post-Harvest Process and Food Engineering, College of Agricultural Engineering, JNKVV, Jabalpur, Madhya Pradesh, India

Mohan Singh
Department of Post-Harvest Process and Food Engineering, College of Agricultural Engineering, JNKVV, Jabalpur, Madhya Pradesh, India

Corresponding Author:
Priti Jain
Department of Post-Harvest Process and Food Engineering, College of Agricultural Engineering, JNKVV, Jabalpur, Madhya Pradesh, India

Process optimization for micro-encapsulation of marigold colorant

Priti Jain and Mohan Singh

Abstract
The present study aims to optimize the dryer parameters (inlet air temperature and feed flow rate) for the process development of marigold colorant from marigold petals extract. A lab spray dryer was employed for the spray drying process. Parameters analyzed were moisture content, solubility, hygroscopicity bulk density, colour, and product recovery. A response surface methodology with three inlet air temperatures (150, 160 and 170 °C) and three feed flow rate levels (80, 120 and 160 ml/h) were investigated. The independent variables significantly affected the physico-chemical properties of the marigold colorant. The optimized condition for spray dried marigold colorant was inlet air temperature of 166.4 °C and feed flow rate of 120 ml/h.

Keywords: Marigold colorant, micro-encapsulation, spray dryer

Introduction

Marigold *(Tagetes erecta* L.) is an important traditional flower crop under cultivation throughout India. In genus *Tagetes* there are 33 species, but commercially two species *T. erecta* and *T. patula* are grown in our country and in western countries another species *T. tenuifolia* is also grown. It is extensively used in religious and social functions in different forms.

Marigold is widely grown in gardens and pots for display purpose. It is also highly suitable for bedding, edging and herbaceous borders. It has great economic potential in loose flower which find industrial application in preparation of natural dyes and essential oils. It is used as mosquito and nematode repellents. It is used for cosmetics because of its amazing virtues for soothing and healing proprieties, and it is a great anti-inflammatory ingredient to deal with your pains.

It has gained popularity amongst gardeners on account of its easy cultivation, wide adaptability and year round flower production. Its free flowering habit, short duration to produce marketable flowers, wide spectrum of attractive colours, shapes, size and good keeping quality has attracted the attention towards it of many amateur and commercial flower growers. Natural pigments offer an alternative to synthetic dyes, but for successful application, an understanding of the chemical and physical properties of the pigment is essential. With the growing legislative restrictions on the use of synthetic colors, a reappraisal of natural plant pigments is taking place with a view to use them as possible colorants in foods. For natural pigments to be accepted as food colorants, legal sanction is a must. With the application of new innovations, natural pigments can become more cost effective and increase their competitiveness against certified dye and dye products. Marigold flowers, which are yellow to orange red in color, are a rich source of lutein, a carotenoid pigment. This pigment has acquired greater significance because of its antioxidant property and for its eye health protection. Although marigold flower extract has been used in veterinary feeds, the potential use of marigold as a natural food colorant has not been exploited to the full extent due to the lack of information on its safety, stability, and compatibility in foods (Sowbhagya et al., 2006) [10] Marigold pigments show good stability to heat, light, pH changes and sulphur dioxide. They are susceptible to oxidation, which can be minimized through encapsulation or the addition of antioxidants such as ethoxyquin, ascorbic acid, tocopherols or butylated hydroxyanisole and butylated hydroxytoluene. Marigold extracts are mixed with a carrier, such as edible vegetable oils, soybean flour, maize flour or distilled water (Setshogo,2005; FDA 2013) [9]. Marigold extracts are used as a yellow to orange colorant in a wide variety of food products including baked goods and baking mixes, beverages and beverage bases, breakfast cereals, chewing gum, dairy product analogues,fats and oils, frozen dairy desserts and mixes, gravies and sauces, soft and hard candy, infant and toddler foods, milk products, processed fruits and fruit juices, soups and soup mixes (Cantrill, 2004) [5]. Fresh and dry flowers are also used to dye wool, silk and cellulose fibres (Setshogo, 2005) [9].

Tagetes erecta flowers are rich in carotenoids and are used to make feed and food pigments. Lutein is the primary xanthophyll pigment that produces the orange colour in marigold flowers, comprising up to 90% of the petals' identified pigments, with smaller amounts of antheraxanthin, zeaxanthin, cryptoxanthin, β-carotene and about 14 other carotenoids (Bosma et al., 2003; Setshogo, 2005) [4, 9]. The marigold flowers are either dried and ground to create marigold meal or extracted with a solvent to produce oleoresins, saponified oleoresins and purified lutein esters (Setshogo, 2005) [9].

The spray dried marigold colorant can be easily added as a natural courant to other foods such as pasta, vegetable oil, margarine, mayonnaise, salad dressing, baked goods, confectionery, dairy products, ice cream, yogurt, citrus juice, and mustard. Thus spray drying is the best alternative to obtain colorants and natural flavouring. Although spray drying of food materials are affected by several parameters but inlet air temperature and feed rate are very important parameters. Therefore, this study was conducted to optimize the dryer parameters (inlet air temperature and feed flow rate) for the process development of marigold colorant from marigold petals extract.

Materials and methods

Preparation of Fresh Marigold Petals Juice

Fresh marigold flowers were purchased from a local market and petals of flowers were removed manually immediately. They were washed in clean water to remove dirt. The juice was extracted by using a juicer. Quantity of extracted juice from one kg of marigold petals was 600ml. The juice was sieved through a sieve with 200 meshes to remove pulp and other solids and then transferred into glass bottles.

Spray drying procedure

A lab spray dryer was used in the spray-drying process. Spray drying was carried out at a various inlet air temperature viz., 150, 160 and 170 °C and feed flow rate viz., 80, 120 and 160 ml/h with constant outlet air temperature of 75 °C and constant air pressure of 2 bar. The total juice solids were adjusted with constant amount 8% of encapsulating agents (maltodextrin). The feed solutions were kept under magnetic agitator at room temperature and fed into the drying chamber through a pump. The dryer was washed with water at the desired setting for 10 min before and after the spray-drying process. Then, spray-dried encapsulated powder was collected, weighed and sealed in plastic container.

Spray Drying of Marigold Petals Juice

Experimental Design

In this study, response surface methodology was used with two factors three level to investigate the individual and interactive effects of drying process variables such as inlet air temperature and feed flow rate on quality attributes moisture content, solubility, hygroscopicity bulk density, colour, and product recovery.

Determination of physicochemical parameters of Marigold Colorant

Moisture content

Moisture content of the sample was determined by using a calibrated Digital Moisture Analyzer (OHAUS, MB45 Moisture Analyzer, New Jersey, UAS) at 100°C. A sample of one g in triplicate was exposed to the heated infra-red coils of the moisture analyzer and the analysis was completed in an automatic mode within 10 minutes.

Solubility

Solubility was determined using a modified version of the method of Al- Kahtani and Hassan, (1990) [3]. A small sample of dry powders of 0.6 g was added to 400 ml of water at 70 °C in a 50 ml beaker. The mixture was stirred using a magnetic stirrer at 7 rpm. Solubility was measured as the time taken to dissolve the dry powders completely.

Hygrscopicity

About 1 g of beetroot powder was spread evenly on Petri dishes (9 cm diameter) to allow for a high surface area between humid air and powder. Samples of each powder were placed in respective seprate dishes in disiccator at 23 °C and 76% relative humidity using HNO_3 solution. Ten minutes interval was selected to get the kinetics of moisture sorption (Al- Kahtani and Hassan, 1990) [3].

Bulk density
Bulk density (g/mL) was determined by gently adding 2 g of powder into an empty 10 mL graduated cylinder. The ratio mass and the volume of powder occupied in the cylinder determine the bulk density value (Goula and Adamopoulos, 2004) [6].

Colour
The color of different samples was measured using a colorimeter (Hunter lab).The results were expressed in b value, which determined the degree of yellowness characteristics of the various marigold colorant samples.

Product Recovery
The product recovery was calculated as the mass ratio of the dried powder collected at the end of spray drying process to the initial solid content in the feed.
The product recovery was calculated with following equation by Ahmad (2019) [2].

$$\text{Product Recovery, \%} = \frac{\text{The weight of obtained spray dried powder}}{\text{The total solid weight in initial feed}} X\ 100$$

Results and discussion
In the present investigation efforts were made to optimize the process parameters for micro-encapsulation of marigold colorant. Effects of process parameters (inlet air temperature and feed flow rate) on physicochemical properties of marigold colorant at constant outlet air temperature of 75 °C and constant air pressure of 2 bar were studied.

Effect of spray drying parameters on physicochemical properties of marigold colorant
The effect of process parameters on the physicochemical properties of the spray dried marigold colorant have been shown in following point:

Moisture Content
The range for moisture content of the spray dried marigold colorant in this study varied from 3.91 to 5.42 %. Thus marigold colorant was quite microbiologically stable. The moisture content of the marigold colorant decreased with the increase in the inlet air temperature due to faster diffusion. From the analysis, inlet air temperature and feed flow rate are the dominating variables and are most significant. Similar results were reported by Quek *et al*. (2007) [8] for the moisture content of the spray dried marigold colorant. Whereas it increased with the increase in feed flow rate, this is mainly due to the fact that during higher feed flow rate, heat transfer between the feed droplets and the drying air became less efficient causing lower water evaporation, thus producing higher moisture content in spray drying process.

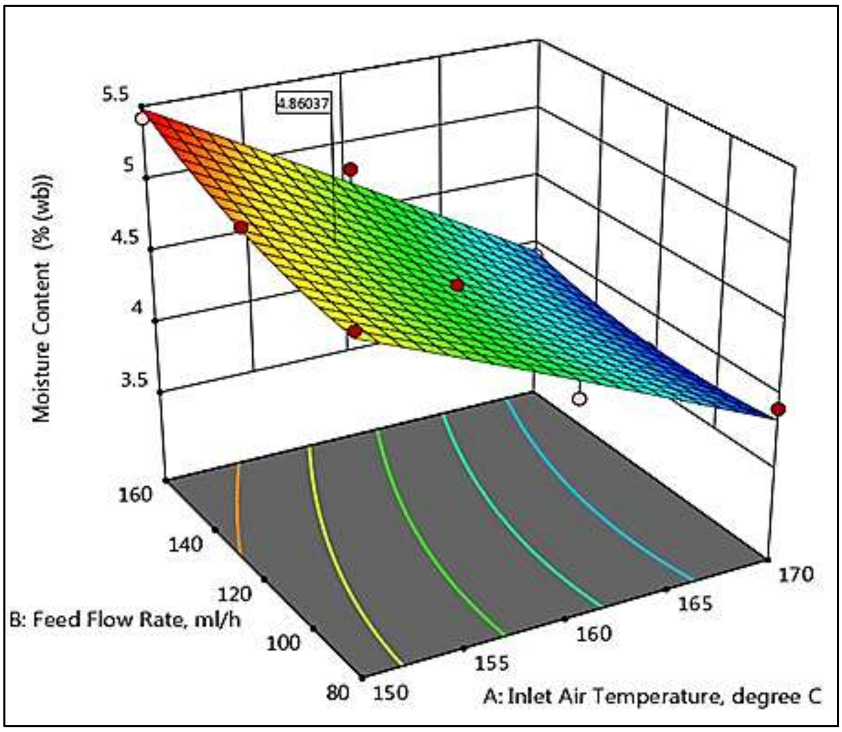

Fig 1: Effect of inlet air temperature and feed flow rate on the moisture content of marigold powder

Solubility
The results showed that solubility of the marigold colorant increased with the increase in the inlet air temperature and decreased with increase in feed flow rate. The range for solubility of the spray dried marigold colorant varied from 81.2 to 95.9 %. Moisture content of the encapsulated marigold powder decreases with an increment in inlet air temperature which results in an increment in the solubility of obtained encapsulated powder. Abadio *et al*., 2004 [1] also reported similar results in case of spray dried pineapple (Ananas comosus) juice powder.

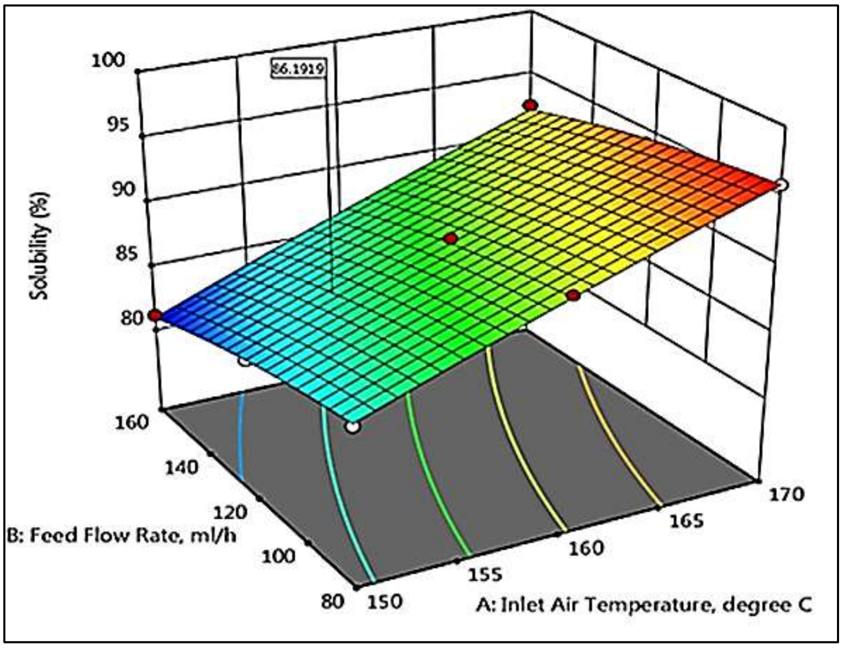

Fig 2: Effect of inlet air temperature and feed flow rate on solubility of marigold powder

Hygroscopicity

In the analysis of hygroscopicity, the graph (Fig.3) indicates that hygroscopicity of the marigold colorant increased with the increase in the inlet air temperature and decreased with increase in feed flow rate. The range for hygroscopicity of the spray dried marigold colorant varied from 0.12 to 0.23 g/g. Less moisture content which causes greater capacity to absorb ambient moisture. Similar results were reported by Quek *et al*. (2007) [8] for the moisture content of the spray dried watermelon powder.

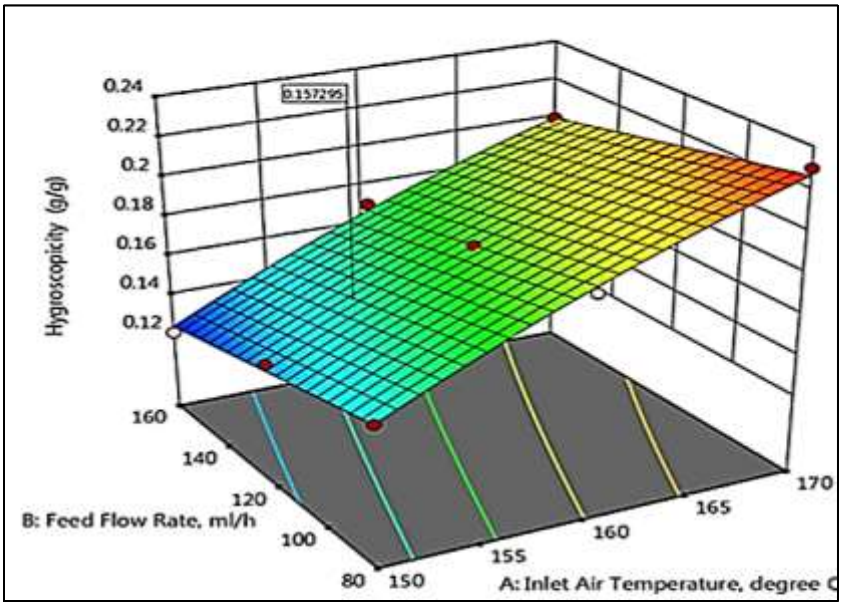

Fig 3: Effect of inlet air temperature and feed flow rate on hygroscopicity of marigold powder

Bulk Density

An inverse relationship between the inlet air temperatures with bulk density was observed. Wheras direct relationship between feed flow rate with bulk density was observed. The range for bulk density of the spray dried marigold colorant varied from 0.32 to 0.47 g/ml. Abadio *et al*., (2004) [1] also reported similar results in case of spray dried pineapple (Ananas comosus) juice powder. This might be due to the lower moisture content of the powders and increase in void spaces. Higher inlet air temperature might have resulted in higher drying rates with less shrinkage of droplets and powder with increased internal voids, so powder had less bulk density.

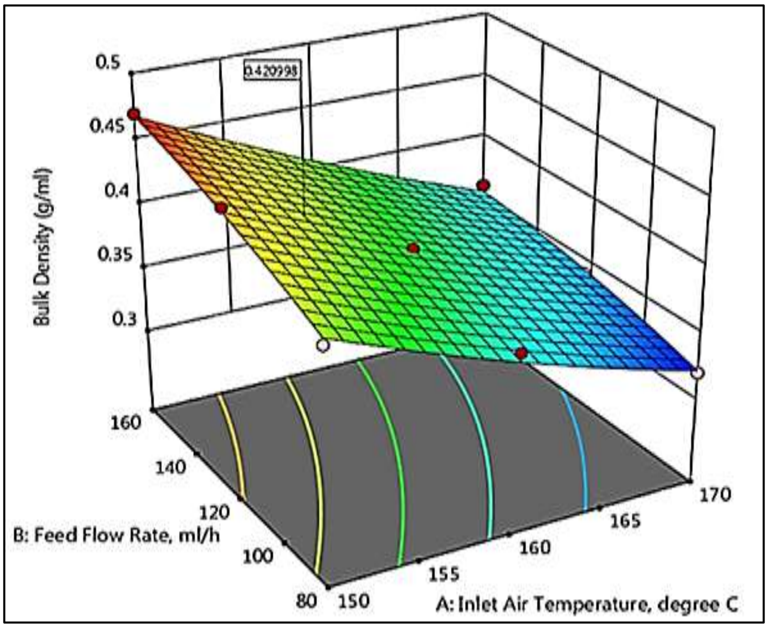

Fig 4: Effect of inlet air temperature and feed flow rate on bulk density of marigold powder

Colour 'b' Value

The combined effect of inlet air temperature and feed flow rate on colour value is shown in figure 5. The range for colour 'b' value of the spray dried marigold colorant in this study varied from 20.12 to 32.27. An increase in inlet air temperature from 150 to 170℃ reduces in colour 'b' value for any constant feed flow rate within the range of 80 to 160 ml/h. This may be due to be the degradation of colour at higher temperature. Whereas an increase in feed flow rate from 80 to 160 ml/h increased colour value for any constant inlet air temperature within the range of 150 to 170 ℃. This may be due to larger amount of residual moisture in encapsulated powder.

Product recovery

The product recovery of encapsulated powder reduced with an increase in inlet air temperature from150 to 170℃ for any constant feed flow rate the range of 80 to 160 ml/h (Fig. 6). It may be inferred that drying temperature produces stickiness problems. Whereas an increase in feed flow rate from 80 to 160 ml/h increased product recovery from for any constant inlet air temperature within the range of 150 to 170℃due to residual moisture in encapsulated powder. The range for product recovery of the spray dried marigold colorant varied from 9.71 to 11.5 %.

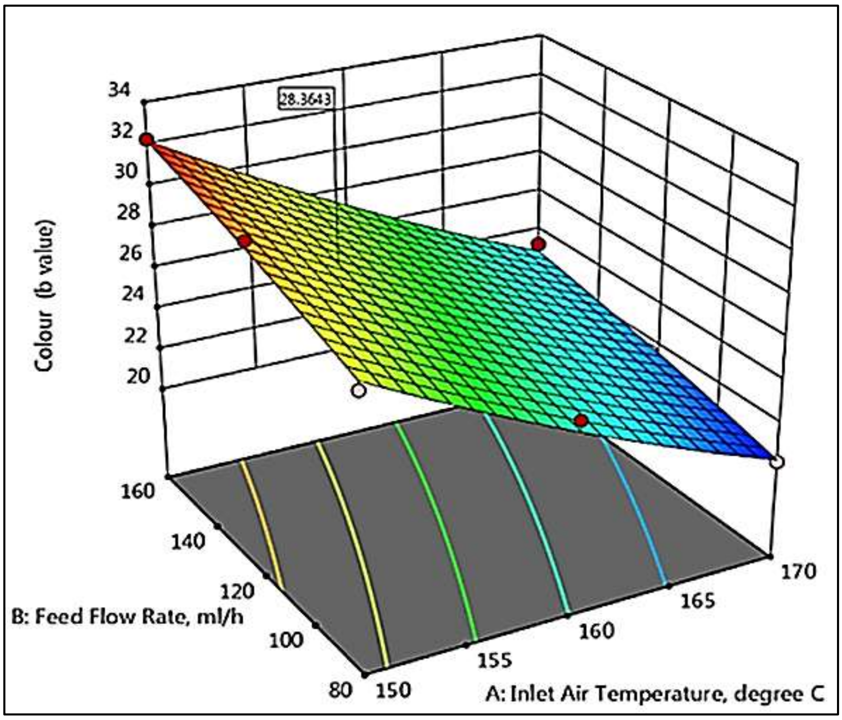

Fig 5: Effect of inlet air temperature and feed flow rate on colour 'b' value of marigold powder

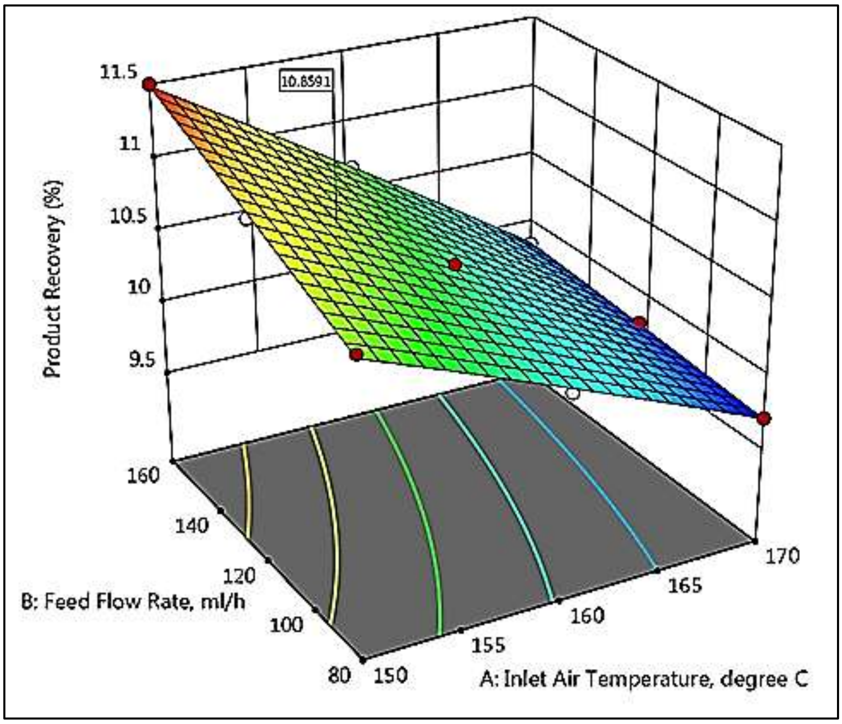

Fig 6: Effect of inlet air temperature and feed flow rate on product recovery of marigold powder

Conclusions

From the obtained results we can conclude that both inlet air temperature and feed rate significantly affect moisture content, yield, solubility, hygroscopicity, colour and bulk density of spray dried marigold extract. Increased feed rate in spray drying caused a significant decrease in solubility and hygroscopicity with high moisture content. colour value, bulk density, and product recovery of marigold powder. Elevated inlet air temperature produced significantly high solubility, hygroscopicity. However, increased inlet air temperature resulted in reduced moisture content colour value, bulk density and product recovery. The optimized condition for spray dried marigold colorant was inlet air temperature of 166.4°C and feed flow rate of 120 ml/h.

Acknowledgement

The authors are grateful to the Jawaharlal Nehru Krishi Vishwa Vidyalaya, Jabalpur, M.P., India for financial grant. We are also grateful to the Department of Post Harvest Process and Food Engineering for their support in carrying out a part of the reported research work. We thank the Department of Food Science for providing lab and instrumental facilities.

Conflict of interest Statement: The authors declare that there is no conflict of interest.

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