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Technology development for the preparation, concentration and utilization of rose extract in different valuable products and by products with retention of color and flavor

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Abstract
Rose is a highly nutritive flower, which find its application in food, medicinal and cosmetic industry. In this study, a process was standardized to produce natural rose preparations and their by-products by utilizing fresh rose petals. The rose extract obtained using 0.5% citric acid was the best in terms of retention of color and fragrance of the natural rose. So this extract was selected for preparation of rose syrup, rose concentrate and by-products such as dried rose residue and rose water. The prepared rose products were found to be rich in phenols, anthocyanins and antioxidants and provided refreshing color and taste of natural fresh rose. Processing of fresh rose petals into different preparations strongly affected the concentration of bioactive components. Slight reductions in bioactive components and total antioxidant activities occurred during storage. Rose syrup prepared from fresh rose petals was found to be highly desirable up to 6 months of storage.

Keywords: Rose petals, syrup, concentrate, anthocyanin, and antioxidants

1. Introduction
The rose plant belongs to the family of Rosaceae and genus *Rosa*, which include 200 different species and more than 18000 cultivars [1]. It has been used for centuries as food components, either in the fresh form or in processed products, such as confectionary and beverages [2]. The total production of rose in India was 19.947 MT [3]. The major rose growing states are Maharashtra, Karnataka, Tamilnadu, Rajasthan, Uttar Pradesh and West Bengal. About 75 per cent of this produce is exported to West-Asian countries in the form of petals, ascertaining the importance of rose for it's by products. Among the various varieties, damask rose (*Rosa damascena* Mill.) is the most important rose species used to produce rose oil, water, concrete and absolute which are valuable and important base materials for the perfume and cosmetic industry [4]. Some varieties are used as landscape plants and among these *desi gulaab,* a red crimson colored rose variety is very common in Punjab.
Rose finds its application in food, medicinal and cosmetic industry. Rose is a highly nutritive flower with a high content of Vitamin C, carotenoids (Vitamin A precursors), phenolic components, some mineral and essential oil [5]. Rose petals have been consumed for many years in cakes, teas and flavor extracts [6]. It has been reported that rose preparations have antioxidant and health promoting activities. Rose preparations are used as astringent, tonic, mild laxative, antibacterial agent and in treatment of sore throat, enlarged tonsils, cardiac troubles, eye disease, gall stones and has been reported to have anti-HIV, anti bacterial and hypnotic activities. The cosmetology use of rose includes use in cosmetic products cosmetic for skin aging, wrinkles and sun damage owing to its antioxidant properties of rose essential oil. The main consumers of rose oil and rose concrete are the big cosmetics and perfumery companies [7].
Rose water which is a by-product of rose is widely used as skin cooling and cleaning agent. In addition to this, dried rose petals are also be used for skin care and the preparation of Gul-e-Roghan for making hair oils. Floriculture is presently considered as the most lucrative agro-enterprise in terms of profit making. As the flowers are the utmost perishable horticulture farm produce, there remains some hindrance in proper marketing following standard post harvest management practices by the common farmers. Hence, value addition by the Argo-industries is another important arena for proper utilization of fresh ornamentals in either garden-fresh or

processed form. Different kinds of value added products are nowadays formulated and marketed by the companies. Some existing rose products in the market are mostly with synthetic colour and flavor like rose lassi (sour milk), rose ice-cream, rose syrup and sweets with rose. These synthetic colors and flavors are sometimes carcinogenic and may cause allergens. For this reason, food and medical industries are increasingly interested in natural sources with high anthocyanin contents for manufacture of supplements with therapeutic and nutritional uses.

Rose preparations could be applied in the food industry as a good source of natural pigments such as anthocyanins due to their attractive color and beneficial health effects and antioxidant activities. Therefore, the present study was undertaken for the purpose of extraction and concentration and utilization of rose preparations in different valuable products and by products.

2. Material and methods

Fresh roses were collected from Shergill Farms, Patiala. The petal were stored at 4˚C prior and used for the analysis of fresh petals and preparation of value added products.

2.1 Preparation of rose products

2.1.1 Rose Syrup

2.1.1.1 Extraction Method: Rose extract was prepared using three solvents i.e. water, alcohol and acid. 50 g fresh rose petals were taken and were grinded with water first. The extract was filtered and separated from the rose residues and again the process of filtering and separating the residue was done. Next extraction was done with alcohol followed by filtering the extract and separating the residue. Last extraction was done with acidified water prepared using 0.5 and 1% citric acid, adding acid into the rose residues again, repeating the steps, combining filtrate so as to obtain a rose extract. It was observed that extracts obtained from water and alcohol were darker and dull in color whereas the rose extract obtained using acidized water containing 0.5% citric acid was the best in terms of retention of color and fragrance of the natural rose. So this extract was selected for preparation of rose syrup.

2.1.1.2 Processing Method: For preparation of 1 liter Rose syrup, sugar 650g, rose extract 250g and water 100ml were mixed thoroughly. The mixture was mildly heated just to dissolve the sugar. Sodium benzoate @ 700 ppm was added to the syrup to extend its shelf life. The prepared syrup was then filled in pre-sterilized bottles and sealed**.**

2.1.2 Dried Rose residue

Rose residue obtained after extraction of rose extract was dried in tray dryer at about 6-7 hours. This residue can also be dried using low cost technologies such as shade drying at room temperature and fan drying**.** Dried rose residue is rich in anthocyanins and can be used in pudding, cakes, *kheer* and *halwa.*

2.1.3 Rose Concentrate

The rose extract was concentrated by rotary evaporator under vacuum (22 mmHg) at 45-50C. The concentration process was continued until the total soluble solids reached to about double the solids of rose extract. Rose water was obtained as a by-product of rose concentration process. Different rose products prepared are shown in Figure 1.

2.2 Quality analyses of fresh rose petals and prepared products

2.2.1 Physico-chemical analysis

The moisture content was determined by drying 5g of fresh tissue in hot air oven at 60±2ºC till constant weight and was calculated on fresh weight basis % [8]. TSS was determined by using refractometer. Acidity, expressed as g citric acid/100g was determined using the titration method with 0.1N NaOH in presence of phenolphthalein as indicator [9].

Fig 1: Different rose products prepared from fresh rose petals

2.2.2 Phytochemical (Bioactive) analysis

Bioactive components including ascorbic acid content, anthocyanins, total phenolic content and antioxidant capacity of fresh rose petals and prepared products were studied. Ascorbic acid content was determined using the dichlorphenol indophenol titration [9]. Anthocyanin estimation was done using the modified method of Tonutare *et al.* [10] and results were expressed as mg/100g. Total phenols were estimated by the Folin-Ciocalteu's colorimetric method [11]. The values were reported as mg of gallic acid equivalent (GAE) by reference to gallic acid standard curve and the results were expressed as mg GAE/100g fw. Total antioxidant activity of the raw tubers was estimated by DPPH (1, 1- diphenyl- 2-picrylhydrazyl) as described by Yamaguchi *et al* [12].

2.3 Sensory quality

Fresh and stored rose syrup was evaluated by a panel of 10 judges using 9-point Hedonic scale for their sensory characteristics like appearance, flavor, texture and overall acceptability. The scores were assigned from extremely liked (9) to dislike extremely (1).

2.4 Statistical analyses

All the experiments were carried out in triplicate. One-way analysis of variance was performed using the SPSS version 20.0 (Statistical Package for Social Sciences). Significant differences
(p<0.05) were determined by Tukey's.

3. Results and discussion

3.1 Physico-chemical and bioactive analysis of fresh rose petals and prepared products

3.1.1 Physico-chemical attributes

The physicochemical characteristics of fresh rose petals and prepared rose products are summarized in Table 1. Significant (p<0.05) differences were found in the moisture content and dry matter content. The moisture content of fresh rose petals was 15.30% and that of products was in the range of 5.03-48.12% (Table 1). Gabriela *et al.* [13] reported moisture

content and dry matter of fresh rose petals as 13.8% and 76.2% respectively. The total soluble solids of fresh petals were 6.0% and it ranged from 15.75-64.30% in prepared products (Table 1). Acidity, expressed as g citric acid/100g varied from 0.008-0.26% (Table 1). The differences observed in Physico chemical attributes of fresh rose petals and prepared products might be due to difference in composition and processing methods.

3.1.2 Phytochemical attributes

Phytochemical attributes i.e. ascorbic acid, anthocyanins, total phenolic content and antioxidant activities measured as DPPH radical scavenging activities of fresh rose petals was significantly (p<0.05) higher compared to processed products (Table 1). The reduction in ascorbic acid and anthocyanins could be attributed to destruction by heat during concentration and drying methods used for processing. Similar reductions in ascorbic acid were reported by Elhadad *et al.* [14] during concentration of apricot and peach juices.

Table 1: Physico-chemical and bioactive compounds of fresh rose and rose products

Parameters	Fresh Petals	Rose Syrup	Rose Concentrate	Rose water	Dry Rose Residue
TSS (^{0}B)	6.0±0.10^d	64.30±0.35^a	45.80±0.25^b	15.75±0.15^c	-
Moisture content (%)	15.30±0.17^d	30.80±0.20^c	32.90±0.25^b	48.12±0.28^a	5.03±0.11^e
Acidity (%)	0.26±0.05^a	0.22±0.04^b	0.20±0.05^b	0.15±0.04^c	0.008±0.01^d
Ascorbic acid (mg/100g)	40.02±0.25^a	6.08±0.11^e	28.18±0.12^b	20.02±0.22^c	15.50±0.15^d
Anthocyanins (mg/100g)	2045.3±2.20^a	1123.0±1.80^c	1943.2±1.50^b	1050.3±2.0^d	1024.3±1.10^e
Total phenols (mg GAE/100g)	348.4±1.40^a	250.8±1.20^c	312.4±1.30^b	212.4±0.08^d	202.5±0.80^e
Antioxidant activity (%)	86.90±0.48^a	63.0±0.25^d	80.1±0.44^b	50.2±0.22^e	68.4±0.21^c

Value within a row with different superscript lower case letters is significantly (p<0.05) different between the different rose products. Mean values ± SD (n=3)

3.2 Storage Studies

3.2.1 Ascorbic acid content

Rose products experienced a significant (p<0.05) decrease in ascorbic acid content during storage. The initial mean ascorbic acid content of different rose products was 17.45 mg/100g, which decreased significantly to 14.88 mg/100g, after 6 months of storage (Table 2). Ascorbic acid is a thermo liable nutrient, which is generally degraded by oxidative processes, which are stimulated in the presence of light, oxygen, heat, peroxidases and enzymes [15]. The ascorbic acid loss accounted for 14.73% after 6 months of storage. Saberian *et al.* [15] observed similar losses in ascorbic acid content during storage of aloe Vera gel at 25°C.

3.2.2 Anthocyanins

During storage, the mean anthocyanin content for different rose products decreased slightly but this decrease was statistically significant (p<0.05) at room temperature (Table 2). This might be due to interaction of ascorbic acid with anthocyanin's which may result in the degradation of both the compounds thorough a condensation reaction [16]. The change in mean anthocyanin content of different rose products during the entire storage period from initial values was only 1.57%. Higher decrease in anthocyanin content was observed by Mgaya-Kilima *et al.* [17] during storage of Roselle-fruit blends at 28°C for 6 months.

3.2.3 Total Phenolic content

During storage, a significant (p<0.05) decrease in total phenolic content was observed in all the prepared rose samples after 6 months of storage. The mean content of total phenolics was estimated to be 244.5 mg GAE/100g initially and this was found to decrease significantly to 221.3 mg GAE/100g after 6 months of storage at room temperature (Table 2). This decrease in phenolics might be due to sensitivity of phenolics to oxidation at above stored conditions. Mean total phenolic content declined by 9.49% after 3 months of storage. Mgaya-Kilima *et al.* [17] reported 58-65% losses in total phenolics of Roselle-fruit blends stored at 28°C for 6 months.

3.2.4 DPPH radical scavenging activity

The radical scavenging activities of prepared rose products were found to decrease significantly (p<0.05) after 6 months of storage (Table 2). The mean radical scavenging activities of rose preparations was estimated to be 65.42% initially and this was found to decline significantly to 63.32% after 6 months of storage. The decrease in the total antioxidant activity may be linked to decrease in the content of phytonutrients such as total phenolics and ascorbic acid. Kapoor and Aggarwal [18] reported significant losses (43.4%) of total antioxidant activity in carrot juice stored at room temperature for 6 months. The correlation between bioactive concentration and antioxidant activity of plant foods is well established [18, 19]. In our study, the loss in mean antioxidant capacity as radical scavenging activity accounted for only 3.20%.

3.3 Sensory quality evaluation of fresh and stored rose syrup

Syrup is a sweet drink made of fruit or medicinal and aromatic plants. Due to a very high sugar content of the syrup in a sealed condition does not require refrigeration and can also be stored longer. The effect of storage on the sensory attributes of rose syrup stored at room temperature (26-38°C/RH 35-87%) is represented in Table 3. Rose syrup prepared from fresh rose petals was found to be highly desirable up to 6 months of storage.

4. Conclusion

The present study was undertaken for the purpose of extraction, concentration and utilization of rose preparations in different valuable products and by products. Rose preparations were found to be rich in anthocyanins which could be successfully applied in the food industry as a good source of natural pigments due to their attractive color and beneficial health effects and antioxidant activities. These natural rose preparations can replace artificial rose flavors present in the market, which are carcinogenic and may cause allergens.

Table 2: Bioactive composition and antioxidant activity of rose products during storage.

Storage months	Rose Syrup	Rose Concentrate	Rose water	Dry Rose
Ascorbic acid (mg/100g)				
0	6.08±0.11^{aD}	28.18±0.02^{aA}	20.02±0.22^{aB}	15.50±0.15^{aC}
2	5.42±0.09^{bD}	27.08±0.21^{bA}	19.18±0.21^{bB}	13.98±0.11^{bC}
4	4.84±0.08^{cD}	25.84±0.14^{cA}	18.42±0.22^{cB}	13.03±0.12^{cC}
6	4.08±0.04^{dD}	25.03±0.12^{dA}	17.98±0.20^{dB}	12.42±0.10^{dC}
Total Phenolic content (mg GAE/100g)				
0	250.8±1.20^{aB}	312.4±1.3^{aA}	212.4±0.80^{aC}	202.5±0.8^{aD}
2	233.4±0.80^{bB}	302.8±0.92^{bA}	205.4±0.70^{bC}	200.8±0.81^{bD}
4	202.5±0.81^{cB}	298.4±0.80^{cA}	200.8±0.72^{cC}	198.2±0.85^{cD}
6	200.2±1.0^{cB}	289.4±0.85^{dA}	200.2±0.82^{cB}	195.4±0.68^{dC}
Anthocyanins (mg/100g)				
0	1123.0±1.80^{aB}	1943.2±1.50^{aA}	1050.3±1.80^{aC}	1024.3±1.10^{aD}
2	1114.5±1.50^{bB}	1935.4±1.80^{bA}	1043.5±1.05^{aC}	1020.2±1.15^{bD}
4	1112.4±1.0^{cB}	1930.2±1.81^{cA}	1038.9±1.12^{cC}	1015.8±1.11^{cD}
6	1110.8±1.10^{dB}	1930.4±1.75^{cA}	1035.1±1.10^{dC}	1010.2±1.12^{dD}
Antioxidant activity (%)				
0	63.0±0.25^{aC}	80.1±0.44^{aA}	50.2±0.22^{aD}	68.4±0.21^{aB}
2	62.5±0.28^{bC}	80.2±0.38^{aA}	49.3±0.22^{bD}	67.3±0.20^{bB}
4	61.5±0.22^{cC}	79.5±0.40^{bA}	48.4±0.18^{cD}	66.8±0.35^{cB}
6	61.1±0.20^{cC}	78.4±0.35^{cA}	47.8±0.20^{dD}	66.0±0.38^{cB}

Values within a column with different superscript lower case letters are significantly (p<0.05) different within the storage duration. Value within a row with different superscript capital letters is significantly (p<0.05) different between the different rose products. Mean values ± SD (n=3)

Table 3: Effect of storage on sensory characteristics of prepared rose syrup

Parameters	Storage Period (Months)			
	0	2	4	6
Appearance	8.32±0.02^{a}	8.30±0.03^{a}	8.20±0.02^{b}	8.20±0.04^{b}
Flavor	8.48±0.03^{a}	8.45±0.02^{d}	8.39±0.03^{b}	8.30±0.03^{c}
Texture	8.41±0.05^{a}	8.40±0.04^{a}	8.38±0.01^{a}	8.24±0.05^{b}
Overall Acceptability	8.51±0.04^{a}	8.46±0.03^{b}	8.45±0.02^{c}	8.35±0.01^{d}

Values within a row with different superscript lower case letters are significantly (p<0.05) different within the storage duration. Mean values ± SD (n=3)

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