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Effect of freeze drying process on keeping quality of static flower using floral freeze dryer

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Abstract
The study was conducted to explore the effect of selected preservation treatments on the quality of Statice flower in floral freeze dryer. Preservation of flowers in freeze drying process retains its pigment (colour) and structural parts (cells) and thus remains as fresh bloom for years. Prior to freeze drying process, flowers were treated with set of chemical compounds for retaining the inherent qualities in preserved flowers. The qualitative characteristics of flowers were evaluated by expert panel. Flowers treated with a chemical compound that had a blend of exchange medium colour preservatives, colour fixatives, buffers, modifiers, pH regulator, and shatter resistant compound (Ethyl vinyl acetate) resulted in flower that was closer to natural.

Keywords: exotic flower, everlasting flower, dehydration technique, drying technology, freeze drying

Introduction
Exotic flowers hold a special place in the hearts of flower lovers due to their breath taking fragrance and exquisite beauty. Exotic flowers are stunning visually and these are used to produce best floral arrangements. Statice, an exotic flower originated in Mediterranean and is used to indicate success. It is a tender perennial with upright sprays of small flowers enclosed in papery calyces, which last for days as a freshly cut flower and for months when dried. They come in bright or pastel shades of yellow, pink, purple, blue, orange, red, and white. The plants have clusters of basal leaves and winged stems. Up to some years ago, they had mainly been grown from seed, but for some time basic material through tissue culture has been available. Fresh flowers may be preserved by drying, but these methods drastically alter the appearance of the flower and increase its frailty. Moreover, dried flowers do not last in areas of high humidity. To preserve the best quality of cut-flowers after harvest and to make resistant to fluctuations in environmental conditions, treatment with floral preservatives is recommended (Zencirkiran, 2010) [7]. Freeze drying flowers technique is an advanced technology and absolutely new to India. For floral preservation in freeze dryer, pre-treatment is recommended. The main advantage of freeze-drying is that it results in products that appear almost like the fresh ones (Nilsback and Zisserman, 2006) [1]. The objective of this study was to explore the effect of preservation treatments on Statice flowers in freeze drying process, using Floral Freeze dryer.

Materials and Methods
Experimental research design was adopted for conducting the study. Violet colour Statice an exotic flower was selected. To produce a high quality freeze dried flower, pre-treatment was found to be necessary. A set of fourteen different chemicals cited in US free Patent 4349459, which fall into the category of exchange medium, biological fixatives, preservatives, environmental fixers, buffers, mordants, pH modifiers, were used in this study and were tested on the flower individually and in combination. Flowers were immersed in each solution for five seconds to study the effect of these chemicals. Each of these chemical solvents was found to play a crucial role on colour, texture, form and appearance of flower. These chemical solvents were blended into different compositions in the on-going Freeze dried flowers research project of the department (Reddy and Kumari, 2010) [3]. Five compositions were selected for use in this research for pre-treatment of flowers. Composition I and II had three solvents having a blend of dehydrant, pigment fixative and colour fixative with pH value of 5-5.5. Composition III and IV had 6 -7 solvents consisting of blend of harsh and mild dehydrant, pigment fixative, colour preservative and pH buffers with pH value between 5.5 – 6.0 and

composition V had all the blend of fourteen solvents consisting of blend of harsh and mild dehydrant, pigment fixative, colour preservatives, environmental and biological fixatives, mordents, modifiers, and buffers with pH of 6.6-7.0. These five basic compositions were tested in Experiment I. To overcome the brittleness and shatter resistance, each of these combinations was blended with a co-polymer and these improvement compositions were used for Experiment II. Each flower was dipped individually in the selected composition and was kept for evaporation in the floral trays before transferring to freezing chamber for dehydration. Each cycle of final experiment in floral freeze dryer took 10-12 days. On completion of freeze drying cycles, flowers were removed from floral freeze dryer and analyzed for measuring the qualitative characteristics such as colour, form, texture and appearance. These four parameters were fixed as variables for evaluation of flowers.

Colour analysis: Statice was photographed and the flower colour was measured before and after freeze-drying using colour Adobe Photoshop (Pantone colour system) and HSB values were recorded. These were used in HSB colour model to explore the change in hue. HSB (Hue, Saturation, Brightness), defines a colour space in terms of three components. Hue (H), the color type ranges from 0 to 360 degree; Saturation (S) i.e. "purity" of the colour ranges from 0 to 100%. The lower the saturation of a colour, the more "grayness" is present and the more faded the colour will appear and Brightness (B) /Value (V), the colour ranges from 0 to 100%.

Moisture analysis: Moisture content and colour of the flower was measured before and after each freeze drying treatment. The percentage of weight loss of flowers was assessed in percentage.

Measurement of qualitative characteristics of flower: Physical Observation tool was developed to observe and to score the freeze dried exotic flowers to assess the effect of different treatments. The following parameters were taken for evaluation which aided in assessing the overall qualitative characteristics of Statice.

Change in Colour in terms of hue, value and intensity
Change in Texture in terms of feel, pliability and suppleness
Form: Change in size, shape, assembly of petals
Appearance: Wrinkle, shrinkage, wilting, fading, deformation.
The three-point scale was used for scoring the variation in each of these qualities. The scores obtained for each of these variables were subjected to analysis to study the effect of treatments on keeping quality of flower.

Results and Discussion

Characteristics of flowers prior to preservation: The fresh Statice flower selected for study was a cluster of calyxes with small flowers which was thick but pliable on the inside and had stiff angular stems in Violet (Mountain's Majesty) colour with the HSB value of H- 281, S- 34 percent; B -74 percent. The flower had soft texture.

Effect of selected solvents on Statice: The effect of 14 chemicals identified in this study, were tested on the flower to observe the reaction of these on colour, texture and appearance of the flower. The observations recorded are presented in Figure 1. Tertiary butyl alcohol has proved to be a harsh dehydrant, causing the flower to turn brittle and turned the violet colour to a darker shade and also luster was lost. 1-propanol was less harsh but turned violet colour to darker shade and petals were pliable and fizzy. When treated with 2-propanol Statice flower retained colour and was closer to natural violet but became coarse and appeared dull.
Sodium phosphate turned violet colour to darker shade and was found pliable and appeared fizzy. The petal treated with Sodium formaldehyde Sulphoxylate was faded and the texture became coarse and wilted. Reaction of Citric acid on Statice had pinkish tinge and texture of the flower became rough and found wilted. Treatment with Thiourea caused fading of colour and became brittle and wilted. Aluminium sulphate had retained the colour but became brittle and luster was lost. A study conducted by Nowack and Rudnicki (1990) [2] said that visual observations during the period of experimentation revealed that the cut roses held in aluminium sulphate retained a desirable freshness of the bloom.
Sodium citrate which is alkaline has proved to be good for this flower as it has retained the natural colour and it was soft, pliable and luscious. With Cupric Sulphate caused the colour to fade and shrinkage and brittle was found. Silicone fluid and Silicone Resin exhibited fading of the colour and wilted but had glossy effect. Treatment with Phenol, had pinkish tinge became coarse and found to be dull, while Propionic acid has turned darker, brittle and wilted.
From the above observations it can be concluded that Aluminium sulphate and Sodium citrate was suitable to retain colour pigments while t-butyl alcohol, 1-propanol and 2-propanol were found suitable for dehydration. As all these characteristics are most desired in dried Statice, it can be inferred that these chemicals have influence on retaining the colour, texture and appearance of the Statice.

Fresh Flower			H- 281 S- 34% B -74% Violet (Mountain's Majesty)
	H-285 S- 37% B-56%		H-287 S-37%, B-60%

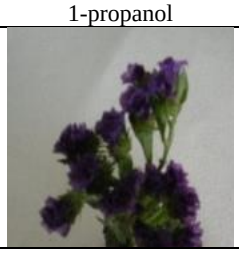
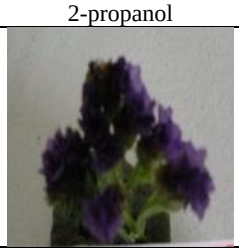
t-butyl alcohol	Violet (Affair)	Aluminium sulphate	Violet (Ce Soir)
	H- 286 S- 45% B-49%		H-278 S-51% B-51%
1-propanol	Violet (Affair)	Sodium citrate	Violet (Eminence)
	H-290 S-50% B- 56%		H-284 S-42% B- 47%
2-propanol	Violet (Affair)	Cupric sulphate	Violet (Honey Flower)
	H-279 S- 46% B- 51%		H-297 S-34% B- 52%
Sodium phosphate	Violet (Affair)	Silicone fluid	Violet (Affair)
	H- 286 S- 49% B-59%		H-279 S-19% B- 46%
Sodium forlamdehyde sulphoxylate	Violet (Studio)	Silicone resin	Violet (Rum)
	H- 289 S- 44% B-60%		H-305 S-34% B- 57%
Citric acid	Violet (Affair)	Phenol	Violet (Violet Blue)
	H- 285 S- 46% B-56%		H-286 S-42% B- 58%
Thiourea	Violet (Affair)	Propionic acid	Violet (Affair)

Fig 1: Effect of solvents on Statice.

Effect of treatment on flowers with basic compositions (Experiment I)

The physical observation scores on different variables identified for studying the effect of different basic treatments on freeze-dried Statice as influenced by different treatments are given in Table 1.

Table 1: Physical Observation score for Experiment I (Basic Composition) of Freeze Dried Statice

Physical Observation Score	Comp-I	Comp-II	Comp-III	Comp- IV	Comp-V
Colour	16	21	15	22	16
Texture	16	15	19	19	20
Form	22	23	23	22	22
Appearance	33	38	34	36	37
Total	87	97	91	99	95

SEm $\pm$ = 1.52

$CD_{0.05}$ = 3.32

Bright Purple colour Statice was tested to explore the suitability of different compositions. The picture of fresh Statice, Statice freeze dried without any treatment, effect of treatments on experiment I and II on freeze dried Statice, the colour variables measured in terms of Hue (H), Saturation (S) and Brightness (B), the name of hue and colour based on HSB values and moisture loss in different treatments are shown in Figure 2. Illustrations presented in this Table for Statice flower with and without treatments, showed the marked variation in terms of colour, form, shape and texture. Freeze dried Statice without any treatment resulted in colour loss, over dried and brittle textured, without lustre, though it retained form. This highlights the importance of pre-treatment for retaining the inherent qualities of flower.

Experiment I with basic compositions showed variations in colour and texture but there were only minor changes in form and shape. Flowers treated with basic composition have retained the natural hue but appeared to differ in their shades. Flowers treated with composition-II and IV appeared closer to natural flower because of presence of Citric acid and Thiourea, while the other three compositions changed the shade of the hue. The main function of the ingredient, Thiourea played a role to prevent the pigment coming out of the plant Wei *et al.* (2010) [6]. A study conducted by Sellegaard (1995) [4] found that the presence of Citric acid provides the plants with a natural colour. It was also observed that irrespective of composition, all the flowers appeared luscious. This proves that chemical used in different composition had influence on the quality of flowers.

Statice treated with shatter resistant polymer in Experiment II, resulted in having all flowers in violet hue, but looked very dull and dryness was more than the above observation. Among these, composition V retained the softness close to reference flower due to the presence of Silicone fluid and Silicone resin. Though Composition I turned darker in colour, flowers were found to be soft and did not shrunk when compared to the other compositions.

In all the treatments the moisture loss percentage varied between 72%-93% in all the flowers and it did not remain consistent between experiments. Improved compositions resulted in excessive dryness in comparison to basic compositions.

From the above analysis it can be noted that colour, texture and moisture content were influenced by the choice of chemicals in both the treatments. The basic composition II and IV was found to produce more natural looking flower. Though composition V in Experiment II was soft in texture, the inherent colour and lusciousness desired in Statice was lacking.

Fresh Flower				**Freeze Dried**
	H -281 S -34% B-74%		H -279 S- 37% B- 57%	
	Violet (Mountain's Majesty)		Violet (Affair)	
Experiment -I			**Experiment -II**	
	H- 265 S- 43% B- 85%	**Comp-I**	H- 264 S- 49% B- 33%	
	Violet (Medium Purple)		Violet (Cherry Pie)	
	M-85%		M-72%	
	H- 280 S- 63% B- 56%	**Comp-II**	H- 263 S- 49% B- 29%	
	Violet (Vivid Violet)		Violet (Violent Violet)	
	M-80%		M-93%	
	H- 288 S- 66% B- 47%	**Comp-III**	H-266 S- 43% B- 27%	
	Violet (Blue Diamond)		Violet (Tolopea)	
	M-78%		M-54%	
	H- 279 S- 57% B- 55%	**Comp-IV**	H- 318 S- 60% B- 34%	
	Violet (Vivid Violet)		Violet(Palatinate Purple)	
	M- 72%		M-60%	

	H- 290 S - 61% B- 30%	**Comp-V**	H- 260 S -34% B -27%	
	Violet (Black currant)		Violet (Jagger)	
	M-73%		M-58%	

Fig 2: Changes in the Physical appearance of freeze dried Statice after Pre-treatments.

Table 2: Physical Observation score for Experiment II of Freeze Dried Statice

Physical Observation Score	Comp-I	Comp-II	Comp-III	Comp- IV	Comp-V
Colour	17	17	13	15	19
Texture	16	15	12	15	21
Form	19	18	15	17	16
Appearance	29	26	25	24	28
Total	81	76	65	71	84

SEm $\pm$ = 1.16
$CD_{0.05}$ = 2.53

Expert's panel scores (Table 2) on qualitative parameters of Statice treated and freeze dried with basic five compositions revealed that scores for the colour, texture, form and appearance was found to have higher score than improved composition. Among the five basic compositions, Composition-II received the maximum score points for overall appearance. However, texture scored least in improved compositions as the flowers were hard and dry.

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