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Original Article

Temperature and Exposure Time Impact on the Extraction of *Opuntia ficus-indica* and *Opuntia dillenii* Cladodes on % Yield as a Response: Screening using Design Expert Software

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ABSTRACT

Objectives: The main aim of the present work is to screen the effect of temperature and exposure time (ET) on the extraction of *Opuntia ficus-indica* and *Opuntia dillenii* cladodes.

Material and Methods: The literature revealed that few attempts were made on the cladodes and other parts of the plants for extracting constituents from them. However, no attempts were made on screening to find out the impact of dependent variables on the independent response. Design of the experiment using quality by design software, namely, Design Expert was used to reveal the impact of the dependent variable on the response.

Results: *O. ficus-indica* and *O. dillenii* were poised, authenticated, and were hauled out with water and the effect of dependent variables (temperature and ET) on the response (% yield) was assessed. The equation coded from this study for the % yield was generated as $+54.30+0.4061A-0.6432B+0.1500AB+0.8375A_2+0.3875B_2$.

Conclusion: The study revealed that 65°C is the optimum temperature and the extraction and ET are directly proportional to extracting the contents from the cladodes.

Keywords: Extraction, *Opuntia*, Response, Screening, Variables

INTRODUCTION

Natural plant substitutes have been extensively studied as an excipient in the pharmaceutical industry for decades.[1] Herbal products are primarily purchased by the pharmaceutical, nutraceutical, food, and cosmetic industries.[2] A lot of demand is there for botanical extracts as well as semi-finished herbal products. Plants of the desert stand out among the cactuses. Cactus has been used in remedies in the past era.[3-7]

All over the world, *Opuntia ficus-indica* breeds in dry and hot weather. It has tall flowers/fruits and this plant is also familiar with the Barbary fig. Therefore, it is well adapted for storing large amounts of rain during irregular rainstorms in dry areas.[8]

Opuntia dillenii is another species of *Opuntia* that grows in the wild in South India. In addition, it has a pear bush and bears fruits around the year. Designed to quickly absorb and store rainwater from uneven rains, it is ideally suited for dry areas.[9] Pads, joints, and cladodes are all common terms for these plants' vegetative parts. The modified stems substitute the leaves in the photosynthetic process and have an ovoid or elongated shape of 18–25 cm in length. The chlorenchyma performs photosynthetic functions. Water is mostly stored in the inner part, which is made up of white medullar parenchyma. When leaves turn into spines, they become fine bristles called areoles. Plants with prickly areoles grow on these rocks. The large ones have sharp edges and are 2 cm long. There are 35 areoles in each internode.[10]

The extracting, phytochemical screening and biological screening of *O. ficus-indica* and *O. dillenii* cladodes have been studied extensively. Furthermore, a comparison of the effects of temperature and time on the % yield was conducted with a factorial design (FD) using Design of Experiment software, despite the previous studies reporting that no investigation has been conducted.

FD

Due to its feasibility to manipulate statistically, traditional research approaches generally study the sway of one variable at a time. Nevertheless, only one factor can be studied at a time. Trying these two factors together leads to false results since they are interdependent. A part of multivariate analysis is the design of experiments (DOE). Nevertheless, DOE is understood to be a treaty with some factors but not all. An objective of DOE is to screen responses and optimize them.[11] The imitations explore possible amalgamations of the levels of the factors. FD's levels are designated as "high" (+1) and "low" (−1), and all input factors are called FD at two levels. The factors for a 2-level design maybe 2, 3, 4, 5, 6, 7, and 8., while the number of runs will be 4, 8, 16, 32, 64, and 128. The number of runs will be extremely augmented above five factors, so fractional FD/or Plackett-Burman design (PBD) is preferred. The screening goal for 2–4 factors is FD, and the response surface goal is the Central Composite Design (CCD) or Box–Behnken design. FD or PBD is an appropriate screening goal for five factors and more, followed by response surface goals. DESIGN EXPERT/STATISTICA/JMP/Unscrambler/Fusion/Minitab is the most widely used DOE software. Basically, the independent variable is entered to create the desired output, called the dependent variable.[12]

MATERIAL AND METHODS

Raw material

O. ficus-indica cladodes and *O. dillenii* cladodes were collected from plants growing in the dry hills surrounding Anantapur, Andhra Pradesh, India. A sample of cladodes measuring 7 × 20 cm was collected. It was identified by the department of Botany of SK University, Anantapur, and authenticated. An Exemplar (SKBD/17/084 and SKBD/17/085) was deposited in the Herbarium.

Extraction of mucilage

For mucilage extraction, cladodes from *O. ficus-indica* and *O. dillenii* were poised and cleaned. Manually separated and crushed medullar parenchyma was unglued from the medullar lining. 250 ml of water was added to 100 g of parenchyma. After stirring at 100 rpm at 50–80°C for 60–90 min, the material was filtered through a domestic sieve (first filtrate). After precipitating with ethanol (95%), the mucilage was recuperated from the filtrate. This resulted in 300 ml of ethanol and was added to 100 ml of the filtrate. The powder mucilage was attained by drying at 50°C for 3 h. A triplicate of each extraction was carried out.[13,14]

RESULTS AND DISCUSSION

As earlier mentioned, that mucilage could be a natural and inexpensive substitute for numerous industrial processes. Therefore, its efficiency should be investigated. Analyses were conducted on different extraction methods. We evaluated the impact of temperature and time on yields.

Mucilage extraction

The mucilage yields from the fresh cladodes using thermal extraction ranged from 50.3–52.5% to 54.1–56.6%, respectively [Table 1]. According to the investigational design data and mucilage % found for each condition, quadratic polynomials were calculated for *O. ficus-indica* and *O. dillenii* cladodes and validated by ANOVA analysis.

Initial risk assessment

According to ICH Q8 and Q9, the Quality Target Product Profile (QTPP) is vital for quality by design (QbD). Moreover,

Table 1: % yields from *O. ficus-indica* and *O. dillenii* cladodes.

Opuntia ficus-indica		*Opuntia dillenii*	
Experiment	**Response (Yield [%])**	**Experiment**	**Response (Yield [%])**
1	51.6	1	55.7
2	52.5	2	56.6
3	50.3	3	54.1
4	51.5	4	55.6
5	51.6	5	55.7
6	52.1	6	56.3
7	51.6	7	56.0
8	50.1	8	54.2
9	50.7	9	54.3

at the beginning of product development, it is important to separate unbiased views from biased ones. The QTPP entails the standards that must be seen for the item to be deemed quality. As a result of past investigations and appraisals of the literature, the QTPP and CQAs for extraction are robust.

Experimental design

In this study, a CCD was adopted for making and assessing quadratic response surfaces for screening the effect of temperature and exposure time (ET) on the extraction of contents from the cladodes of *O. ficus-indica* and *O. dillenii*. Stat-Ease Inc. used Design-Expert software (11.0) to assess the parameters. With the resultant quadratic model, the key, interface, and quadratic properties of independent variables on dependent variables were measured as $Y = B_0+B_1X_1+B_2X_2+B_{12}X_1X_2+B_1X_{12}+B_2X_2^2$.

Here, Y is the dependent variable, B_0, B_1, and B_2 are the regression coefficients of independent variables and their mutual interactions, and X_1 and X_2 are the independent variables (temperature and ET). A dependent variable/ response was the %yield of *O. ficus-indica* and *O. dillenii* cladodes. The variables and their levels used in the screening of the impact of independent variables on the response are exemplified in [Table 2] and central composite design experimental designs are expressed in [Table 3].

The response surface methodology (RSM) was used to test the influence of variables (time and temperature) on extraction. As the maximum values of the response surface of the temperature and time curves, the optimum conditions for extracting mucilage were found. The untried designs were created and estimated with Stat-Ease-Design ExpertV.11. Statistical validation was performed using a one-way analysis of variance ANOVA with a confidence level of 95%. The models gained were based on a constrained central composite design constructed from a factorial design and star points. The response from the study was expressed as Yield (%) = $+50.70+0.3509A-0.5527B+0.0750AB+0.6063A^2+0.1062B^2$.

In terms of coded factors, the reckoning can be used to envisage the response to given levels of each influence. By default, the factors with high levels are coded as +1 and those with the low levels are −1. When the coefficient of the factors is compared, the coded equation can show which factors have the greatest impact. Fit summary for the response of %yield of *O. ficus-indica* and *O. dillenii* cladodes was suggested Quadratic and Linear models [Tables 4 and 5].

In addition, the ANOVA for the Quadratic model for the response (% yield) of *O. ficus-indica* and *O. dillenii* cladodes is shown in [Tables 6 and 7].

10.59 is a significant F-value for the model. The probability of an F-value this large occurring due to noise is as low as 4.01%. Model terms are significant if their *P*-values are

Table 2: Factors and their levels used in the study.

Factor	Levels		
	−2	0	+2
1 (A): Time (min)	60	75	90
2 (B): Temperature (°C)	50	65	80

Table 3: The experimental design by Design-Expert software for studying the responses for the extraction of contents from the cladodes of *O. ficus-indica* and *Opuntia dillenii*.

Trials	Factor 1 (A) Time (min)	Factor 2 (B) Temperature (°C)
1	60	50
2	90	50
3	60	80
4	90	80
5	53.7868	65
6	96.2132	65
7	75	43.7868
8	75	86.2132
9	75	65

Table 4: Fit summary for the response %Yield of Opuntia ficus-indica cladodes.

Source	Sequential *P*-value	Adjusted R^2	Predicted R^2	
Linear	0.0387	0.5492	0.2633	Suggested
2FI	0.8088	0.4659	0.1104	
Quadratic	0.0644	0.8570		Suggested
Cubic	0.3354	0.9517		

Table 5: Fit summary for the response (%Yield) of *Opuntia dillenii*.

Source	Sequential *P*-value	Adjusted R^2	Predicted R^2	
Linear	0.0445	0.5276	0.3278	Suggested
2FI	0.6861	0.4532	0.2424	
Quadratic	0.0442	0.8861		Suggested
Cubic	0.1278	0.9944		

Table 6: ANOVA for quadratic model for the response 1 (% yield) *Opuntia ficus-indica*.

Source	Sum of squares	F-value	*P*-value
Model	4.90	10.59	0.0401
A-Time	0.9850	10.64	0.0471
B-Temperature	2.44	26.39	0.0143
AB	0.0225	0.2430	0.6559
A^2	1.07	11.55	0.0425
B^2	0.0328	0.3547	0.5934
Residual	0.2778		
Cor total	5.18		

<0.0500. Model terms A, B, and A^2 are significant here. Model terms are not significant if the value exceeds 0.1000. The reduction of model terms may improve your models if there are a lot of insignificant terms (except those that are required for hierarchy support).

This indicates that the model is significant with an F-value of 13.45. An F-value of this size is only likely to occur due to noise by 2.88%. If the P-values for model terms are <0.05, then they are significant. A, B, and A^2 are significant model terms. If the value is >0.1001, then the model terms must not be significant. It may be beneficial to reduce model terms (excluding those necessary to support the hierarchy) if you have many insignificant variables. The % yields from *O. ficus-indica* and *O. dillenii* cladodes are embodied in [Table 1].

Table 7: ANOVA for quadratic model for the response (% yield) for *Opuntia dillenii*.

Source	Sum of squares	F-value	*P*-value
Model	6.86	13.45	0.0288
A-Time	1.32	12.92	0.0369
B-Temperature	3.31	32.42	0.0107
AB	0.0900	0.8816	0.4170
A^2	2.04	19.99	0.0209
B^2	0.4368	4.28	0.1304
Residual	0.3063		
Cor total	7.17		

For fresh cladodes, the linear term for time has a positive coefficient, indicating that mucilage extraction increases over time. A minimum in the response surface indicates that large variations in time in either direction result in increased extraction yields (positive quadratic terms). While the linear term for temperature does not affect mucilage yield, the interaction between the two variables determines the extent to which mucilage can be extracted. Large temperature variations either in the positive or negative direction increase extraction yield, as indicated by the quadratic terms. During the time range considered (50–80 min), mucilage extraction appears not to be significantly affected by time. Taking the confidence intervals into account, the interactions between variables have no significant outcome on mucilage withdrawal. The interaction effect of temperature and ET on the % yield from the cladodes of *O. ficus-indica* and *O. dillenii* is expressed in [Figure 1], whereas the contour plot and 3D response plots are represented in [Figure 2].

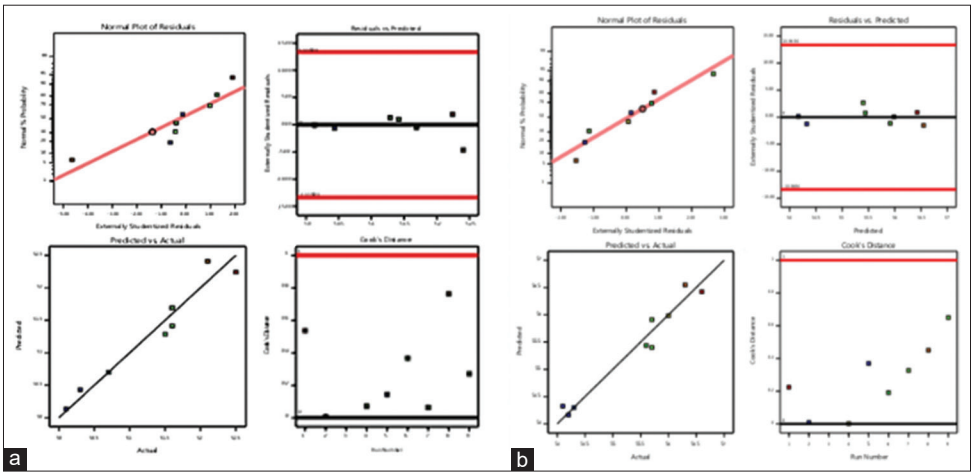

Figure 1: Plots showing the interaction effect of temperature and exposure time on the % yield from the cladodes of *Opuntia ficus-indica* and *Opuntia dillenii*.

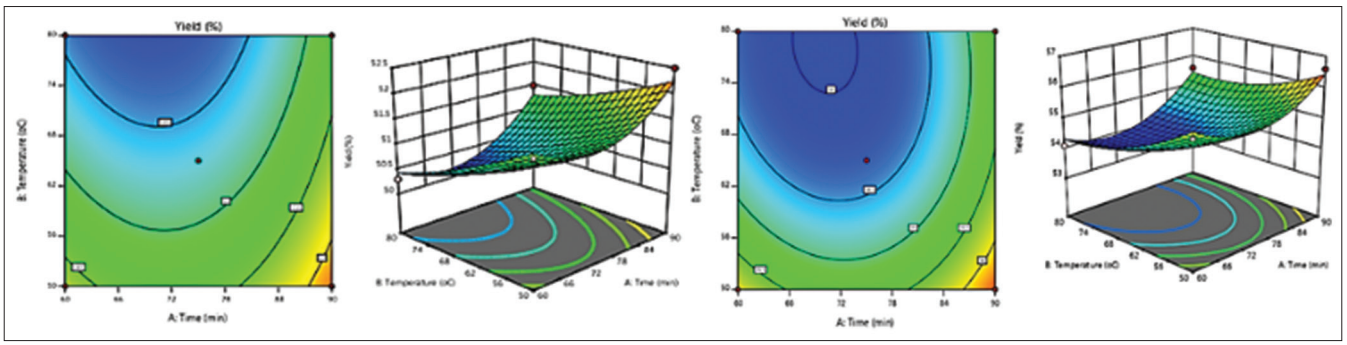

Figure 2: Contour plot and 3D response plot for the response (%yield) from *Opuntia Ficus indica* (Left) and *Opuntia dillenii* (right).

RSM was used to test the influence of variables (time and temperature) on extraction. We took the effects of temperature and time on yield as independent variables. A response surface whose maximum value represents the optimum conditions for extracting mucilage was attained. ANOVA with a 95% confidence level was used to generate and evaluate the experimental designs using Stat-Ease-Design ExpertV.11. Based on a factorial design with few star points, we generated models with circumscribed central composite designs. The final equation in terms of coded factors was: Yield (%) = +54.30+0.4061A–0.6432B+0.1500A B+0.8375A_2+0.3875B_2

The equation in the form of coded factors can be used to envisage the retort at dissimilar levels of each factor. By default, the high values of the factors are coded as +1 and the low values as −1. Through the comparison of factor constants, the coded equation can be used to determine how much each factor contributes to the outcome.

CONCLUSION

The thorough work in finding the impact of temperature and ET on the extraction of *O. ficus-indica* and *O. dillenii* cladodes, the design of the experiment using QbD software, namely, Design Expert was used. The study concludes that a temperature of 65°C is optimum, and the ET is directly proportional to extracting the contents from the cladodes. The equation derived from the study (impact of temperature and ET on the % yield) was found to be +54.30+0.4061A–0.6 432B+0.1500AB+0.8375A^2+0.3875B^2.

Declaration of patient consent

Patient's consent not required as patients identity is not disclosed or compromised.

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Nil.

Conflicts of interest

There are no conflicts of interest

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