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FORMULATION AND CHARACTERIZATION OF LAKE COLOR OBTAINED FROM BLACK CARROT

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ABSTRACT: Colorants are mainly used to impart a distinctive appearance to the pharmaceutical dosage forms. The present study was carried out to develop aluminium lake color of a dye obtained from black carrot using different adsorbents (Aluminium oxide or aluminium hydroxide) in different ratio by simply mixing them to get dye adsorbed onto the surface of adsorbent. Adsorption experiment was carried out for 60 minutes. Aluminium oxide at a concentration level of 30% w/v found to be the choice of adsorbent after optimization because maximum adsorption of dye from reaction mixture after 60 minutes and maximum % yield. After optimization of adsorbent the effect of pH and temperature also studied by formulating lake by varying these two parameters. It was observed that a higher temperature (50°C) and a lower pH (3) favor the adsorption. The lake then characterized for various physicochemical properties like angle of repose, Carr's index, hausner's ratio, loss on drying, particle size and limit test for heavy metals. The lake was found to follow pseudo second order kinetics.

INTRODUCTION: Coloring may be required to increase the aesthetic appearance or to prolong the stability or to produce standard preparations or for identification of a particular formulation. The prime priority of colorants is to increase the aesthetic appearance of the product, so we can say that the colorants are the cosmetics for the pharmaceutical formulations. Pharmaceutical preparations are colored mainly for following reasons: to increase acceptability, for identification, making standard preparations, and for increases stability of the formulation.

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Natural Food Coloris are any dye, pigment or any other substance, obtained from vegetable, animal, mineral or source capable of coloring food, drug, cosmetic or any part of human body. Natural colors obtain from variety of sources such as seeds, fruits, vegetables, algae & insect. Lakes have been defined as the Aluminum salts of water soluble dyes extended on a substratum of alumina. The method of preparation of the alumina hydrate and the conditions under which the dye is added or absorbed determines the shade, particle size, dispersability as well as tinctorial strength.

Other important variables are the temperature, concentration of reactants, final pH, and the speed and type of agitation. The shade or hue of a lake varies with the pure dye content. Aluminium lakes are prepared under aqueous conditions by reacting Aluminium oxide with coloring matter complying with purity criteria set out in the appropriate specification monograph. Following lake formulation, the product is filtered, washed with water and dried. [1, 2]

MATERIAL AND METHODS:

Aluminium oxide (Thomas Baker- Mumbai), Aluminium hydroxide gel dried (Thomas Baker- Mumbai), Sodium hydroxide pellets (Sisco Research laboratories Pvt. Ltd. Mumbai), Potassium-di-hydrogen ortho-phosphate (Sisco Research laboratories Pvt. Ltd. Mumbai), Hydrochloric acid (Thomas Baker- Mumbai) and Buffer tablets (pH 7) (Himedia laboratory, Mumbai) were of laboratory grade, provided by pharmaceutics laboratory, BN PG College, Udaipur. The vegetables for color extraction i.e. black carrot was purchased from Reliance Fresh, Udaipur and Reliance Fresh, Jaipur.

Reagent Preparation:

0.1 N NaOH, 0.1 N HCl, 0.1 N Phosphate buffer pH 7 was prepared.[3]

Preparation of Standard Curves:

Black carrot dye was estimated at the λ_{max} at various pH using water as media obeyed Beers law.

λ_{max} at various pH are as:

FIG. 1: ADSORPTION EXPERIMENT

	Black Carrot		
	pH 3	pH 6	pH 8
λ_{max}	521	521	558

Adsorption Experiment: [4]

Adsorption experiments were carried out by agitating adsorbents (aluminium oxide or aluminium hydroxide or aluminium oxide+ aluminium hydroxide) with dye solution of desired concentration and pH in a 100 ml beaker at desired temperature. A good contact has been made between adsorbent and dye by agitating on hot plate magnetic stirrer of cosolab at low rpm in a 100 ml round bottom flask. Dye concentration remained in the solution was determined spectrophotometrically by monitoring the absorbance at desired λ_{max} using single beam UV-VIS Spectrophotometer. Experiment was continued to 1 hr and 2 ml of each sample was withdrawn at 10 minutes time interval (In sample tube) replacing with the same amount of distilled water. All the samples were then centrifuged for 20 minutes at medium rpm. One ml of the supernatant solution was diluted to 5ml and analyzed to get UV absorbance at its respective λ_{max} while distilled water was used as blank.

Experimental Design: [5]

A 3^2 full factorial design was employed to systematically study the combined influence of the effect of independent variables (Adsorbent type- Aluminium oxide and Aluminium hydroxide) on the dependent variables i.e. % adsorbed. In this design 2 factors are evaluated, each at 3 levels, and experimental trials are performed at all 9 possible combinations.

TABLE 1: FACTORIAL DESIGN FACTORS AND THEIR LEVELS FOR ADSORBENTS

Aluminium Oxide			Aluminium hydroxide		
Medium (0)	High (+1)	Low (-1)	Medium (0)	High (+1)	Low (-1)
20%	30%	0%	20%	30%	0%

TABLE 2: FACTORIAL DESIGN FACTORS AND THEIR LEVELS FOR PH AND TEMPERATURE

pH			Temperature (°C)		
Medium (0)	High (+1)	Low (-1)	Medium (0)	High (+1)	Low (-1)
3	8	6	30	50	40

After optimizing adsorbent type and its level again a 3^2 full factorial design was employed to systematically study the combined influence of the effect of independent variables of Batches (pH and temperature) on the dependent variables % adsorbed. In this design 2 factors are evaluated, each at 3 levels, and experimental trials are performed at all 9 possible combinations. A statistical model incorporating interactive and polynomial terms is used to evaluate the response.

Kinetics Study: [6]

The data from one hour adsorption study were fitted into three kinetics models:

Pseudo-first-order rate equation of Lagergren

$$\log(qe-q_t)= \log qe- k_t/2.303 * t$$

Pseudo-second-order rate equation

$$t/q_t=1/k_2qe_2+1/ q_e*t$$

The intraparticle diffusion model

$$q_t=k_tt^{1/2}+C$$

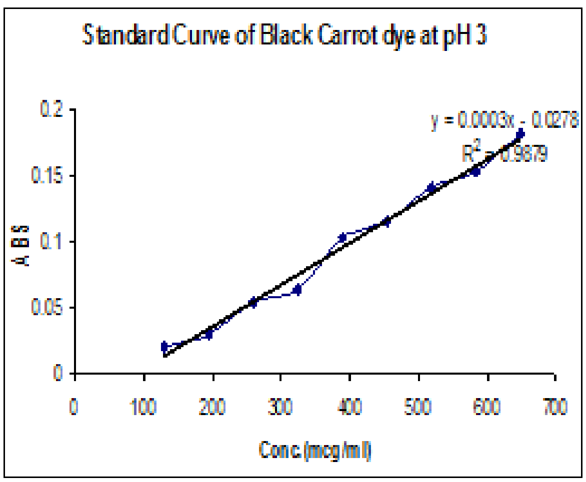
Where q_e and q_t are the amounts of the dye adsorbed (mg) at equilibrium and at time t (min), respectively. $k_1$is the adsorption rate constant (L min^{-1}) for 1st order kinetic, k_2 (g $mg^{-1}min^{-1}$) is the rate constant of pseudo-second-order adsorption. k_t($mgg^{-1}min^{-1/2}$)is the intra particle diffusion rate constant.

Physicochemical Characterization:
The optimized lake was chacterized for various physicochemical properties like angle of repose [7], loss on drying [3], carr's index [8], hausner's ratio [8], organoleptic properties and limit test for heavy metals [9].

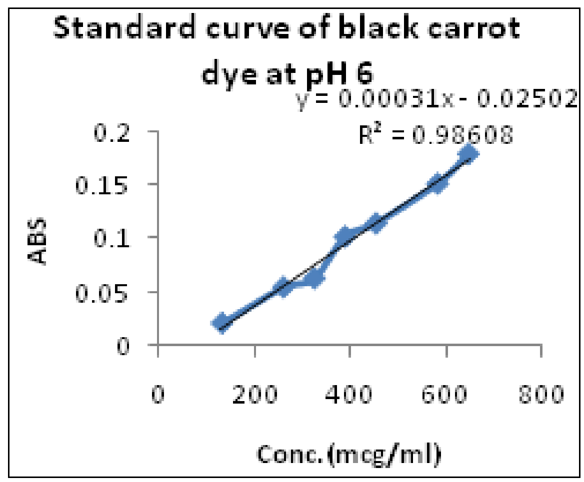
Stability Studies:
The optimized formulation after characterization were subjected to stability studies under normal conditions temperature and humidity in desiccators for three months in packed in aluminium foil to protect it from any sunlight. After three months the lake were assayed and compared from the initial product for its specific color.

RESULTS AND DISCUSSION:

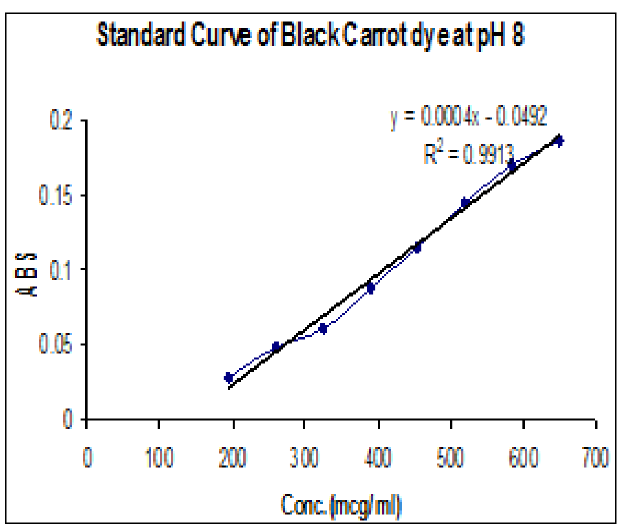
The standard curve obtained as follows:



2A: At pH 3



2B: At pH 6



2C: At pH 8

FIG.2: (A, B, C) STANDARD CURVES

Black carrot color extracted from black carrot and lake color were formulated using aluminium oxide or aluminium hydroxide or in combination of these two adsorbents at various concentration. According to factorial design eight batches from F1 to F8 were formulated and % dye adsorbed (By UV spectrophotometry) and % yield were calculated at every 10 minutes till one hour. The Adsorption data are shown in **Table 3** and adsorption profiles are in **Fig.3.**

TABLE 3: ADSORPTION PROFILE OF BLACK CARROT DYE BY VARIOUS ADSORBENTS

	% Adsorbed at various time interval (Minutes) *					
Batches	**10**	**20**	**30**	**40**	**50**	**60**
F1	75.65±0.172	80.48±0.29	83.40±0.17	86.16±0.00	86.50±0.172	86.68±0.00
F2	76.34±0.17	81.51±0.17	84.61±0.172	87.19±0.00	87.19±0.17	88.23±0.172
F3	79.96±0.298	86.16±0.3	92.02±0.45	93.91±0.00	94.26±0.17	94.77±0.172
F4	80.30±0.172	83.92±0.17	86.68±1.033	91.33±0.298	92.53±0.172	92.71±0.17
F5	79.10±0.17	85.99±0.17	91.16±0.29	92.71±0.172	93.40±0.3	93.74±0.00
F6	79.27±0.172	82.71±0.17	85.64±0.30	90.47±0.172	91.33±0.29	91.67±0.172
F7	80.65±0.62	87.54±0.17	92.88±0.298	94.95±0.3	97.01±0.29	97.36±0.17
F8	79.79±0.172	85.82±0.17	91.33±0.00	93.22±0.17	94.43±0.3	94.95±0.29

.*Values showed % adsorbed ± SEM (n=3)

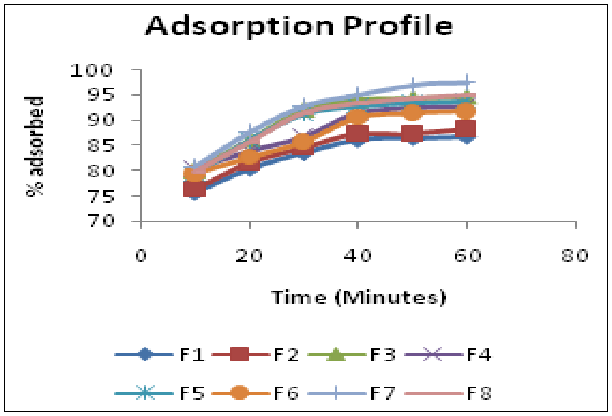


FIG. 3: ADSORPTION PROFILE OF VARIOUS BATCHES

The batch F7 (adsorbent aluminium oxide at 30% w/v concentration) was found to be the best batch as it showed maximum adsorption of the dye. Further this batch was studied for effect of pH and temperature.

Effect of Temperature and pH:

Once the suitable adsorbent for black carrot dye and its concentration was optimized for lake formulation, a series of experiments of lake formulation were conducted at different temperature (30, 40 and 50^{0}C) and various pH (3, 6 and 8) while keeping the adsorbate and adsorbent concentration, contact time, agitation speed constant. The batch F17, the lake formulated at 50^{o}C and pH 3 showed maximum adsorption of dye and % yield as in the **Table 4** and **Fig. 4.**

TABLE 4: ADSORPTION PROFILE OF BLACK CARROT DYE AT VARIOUS pH AND TEMPERATURE

	% Adsorbed at various time interval (Minutes) *					
Batches	**10**	**20**	**30**	**40**	**50**	**60**
F11	75.19±0.3	82.42±0.12	90.47±0.17	93.47±0.21	95.99±0.00	96.21±0.00
F12	62.10±0.2	69.81±0.19	72.77±0.32	79.61±0.50	82.14±0.12	85.32±0.2
F13	52.74±0.45	60.83±0.52	67.55±0.3	70.48±0.62	77.55±0.46	78.41±0.29
F14	68.26±0.3	73.19±0.49	80.31±0.11	85.17±0.26	87.79±0.32	90.27±0.18
F15	65.99±0.33	72.11±0.46	74.39±0.59	80.19±0.61	84.22±0.11	88.14±0.0.29
F16	59.11±0.61	65.56±0.21	69.9±0.45	73.51±0.46	78.7±0.42	80.21±0.3
F17	77.44±0.11	84.88±0.26	91.00±0.44	93.72±00	96.79±0.41	98.79±0.21
F18	70.29±0.51	78.1±0.21	82.37±0.22	90.17±0.52	92.33±0.3	93.22±0.41
F19	61.24±0.34	67.92±0.42	70.11±0.39	77.24±0.61	81.33±0.29	84.99±0.3

.*Values showed % adsorbed ± SEM (n=3)

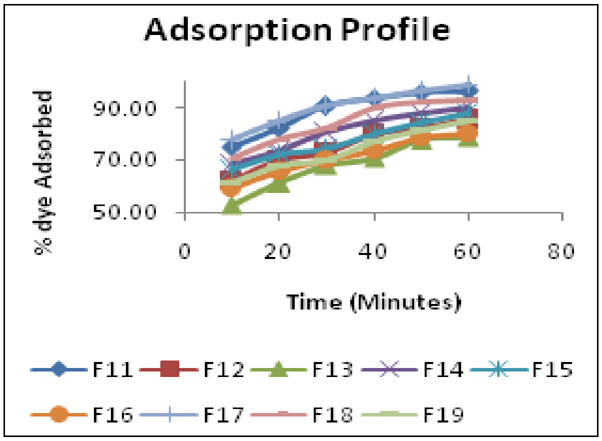
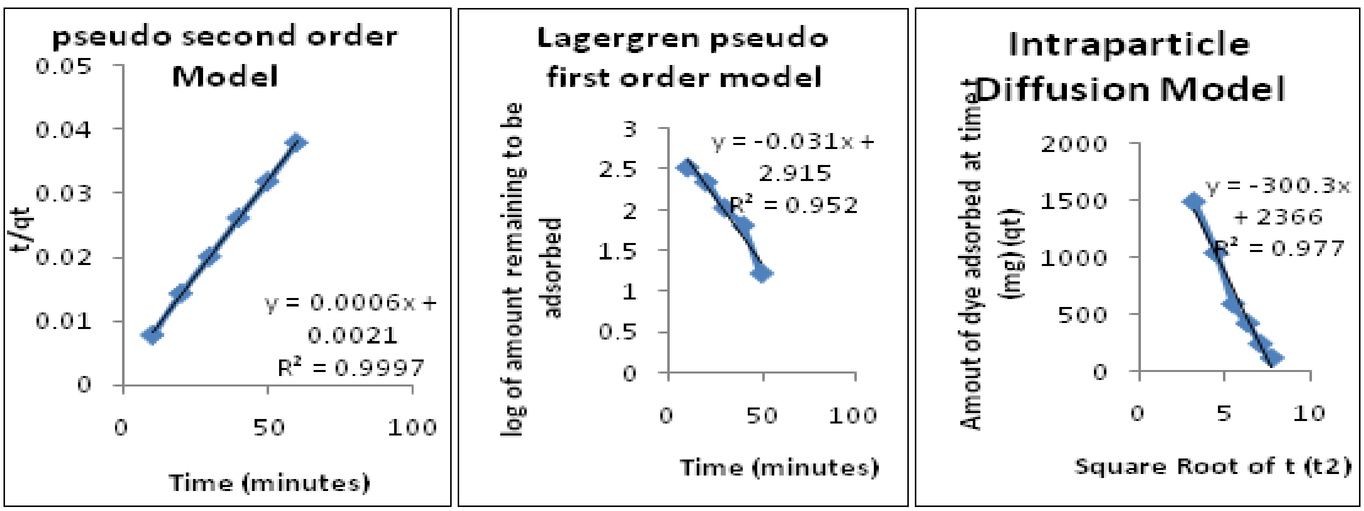


FIG.4: ADSORPTION PROFILE OF VARIOUS BATCHES

Kinetics Study:

The kinetics study of lake formulated from batch F17. The data obtained was fitted into three models pseudo-first-order (Langergren), pseudo-second-order (Ho and McKaY) and the intraparticle diffusion model to find the type of kinetics of adsorption.



5A: PSEUDO SECOND ORDER **5B: LAGERGREN PSEUDO SECOND ORDER** **5C: INTRAPARTICLE DIFFUSION**

FIG.5: (A, B, C) KINETICS STUDY ACCORDING TO THREE MODELS

The kinetic parameters for three kinetic models and correlation coefficients were calculated from the plots (**Fig. 5**). From this data it was observed that the adsorption followed pseudo second order reaction kinetics having a greatest r^2 value = 0.9997.

Physicochemical Characterization:

TABLE 5: PHYSICOCHEMICAL PROPERTIES

Batch	Angle of Repose(θ)*	Tapped Density (gm/cm^3)*	Bulk Density (gm/cm^3)*	Hausner' ratio*	Carr's Index* %
F17	20.18±0.17	0.45±0.01	0.38±0.00	1.18±0.122	18.42±0.142

*Values showed average±SEM(n=3)

Limit test for heavy metals was found to be in limit. Loss on drying was 0.057± 0.02 to 0.1552 ±0.041 i.e. in the limit of loss on drying according to IP. The lake colors obtained from black carrot dye were of light purple to purple in color and having a vegetable odor with a smooth texture. Particle size ranges from12.78 µm to 2.84 µm having a particle size of 7.44 µm.

SUMMARY AND CONCLUSION: The present study was aimed to formulate Aluminium Lake of black carrot dye and its characterization. It is found that this lake is best formulated when aluminium oxide alone is used as an adsorbent rather than using aluminum hydroxide or a combination of aluminium oxide and aluminum hydroxide. Adsorption is a surface phenomenon so depends upon concentration of reactant. If there is a concentration of aluminium oxide is high as upto 30% w/v there is a better adsorption than lower ranges. Temperature and pH also the major factors since temperature is related with the activation energy and pH with the ionic state of the pigment. Itwas concluded that a higher temperature and a lower pH value favors the adsorption process.

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