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Adsorption of Thymol Blue and Erythrosine-B on MWCNTs Functionalized by N-(3-nitrobenzylidene)-N'-trimethoxysilylpropyl-ethane-1,2-diamine Equilibrium, Kinetics and Thermodynamic Study

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ABSTRACT

We have accomplished a synthesized of an adsorption material through anchoring N-(3nitro-benzeyliden)-N-trimethoxysilylpropylethan-1,2-diamin onto multi-wall-carbon nanotube. The systems were established via SEM and FT-IR techniques. Then, it was used for removing the ultra-sound-assisted for Thymol Blue (TB) and Erythrosine-B (EB).The pH dependency and initial dyes concentration, sonication time and adsorbent's dosage for the removing percentage of Thymol Blue and Erythrosine-B which have investigated by CCD or Central-Composite Designing. It was exhibited that the Thymol Blue and Erythrosine-B adsorption follows, while the Langmuir models explain the equilibriums variables.

Keywords: Multi-walled carbon nanotubes**,** Surface methodologies, Ultra sounded-assisted dye removing, Thymol Blue (TB) and Erythrosine-B (EB)

INTRODUCTION

Dyes and pigments have been released in the waste-waters from textiles, papers, rubbers, plastics, leathers, cosmetics, foods, and drug industries[1]. These dyes make allergic dermatitis[1], skins irritation, cancers and mutations in living[1,2] organism. It also causes eyes burn, which might be reason for permanent injuries to the eye's human and animal also.. Discharge[1,2] of a dye into waters sources threaten the water supplies and qualities due to irr-efrangible[2] under harshest condition, toxicities, accumulations and magnifications throughout[3] the foods chain[2,3]. The high colored's effluents not only cause damages for living but human being via producing mutagenic[3] effects[3].

Dye generally has a complex aromatics structure which makes them stables and difficulty for decomposing. The dye presents in wastewater[3] absorbs sunlight's leading to decreasing in the efficiencies of photo-synthesis in the aquatic plant due to reducing lights penetration[4].

Among various types of dye including anionic[4], cationic[5] (basically dye), and non-anion (disperse[5] dye), the positive ion is the most toxic dyes[5]. Among various methods of dye's removal from aqueous solutions such as membrane[6] separation[6] are important. For the chemical oxide materials the adsorption techniques are known as an important methods for dye removal[7,8].

Thymol Blues, belongs to tri-phenyl-methane[7] groups, have biological activities same stains agents. The TB[8] dyes are also used for pH indicators in analytical chemistries. Inhalation leads to respiratory[8] tract exposures and subsequently[8]. Skin Contacts may causes irritations with redness[8] and pain[9].

Erythrosine-B is a water-soluble xanthenes class of dye. It is extremely used as colorant[9] in foods, textiles, drugs and cosmetics[10]. In high dose it cause several types[9,10] of allergies[10], thyroid activity, carcinogenicities, DNA damages, and neurotoxicity's behavior in the human[9] and animal. The photo-chemicals and bio-chemicals degradations of the erythrosine[10] are not suggested because for formation toxic by-products [10]. Adsorptions are the best techniques[11] for removing of toxics and noxious[12] impurity in comparison[12] to other conventional[13] protocols same as chemical coagulation[12,13], ion exchanges, electrolysis of electrolytes, biological[13] treatment which are related to advantages[14]. Adsorption technique[12] also have a lot of efficiencies for the removing pollutant which are highly stables in biological degradations process through economically[14] mild pathways[11-13]. Of those methods[14], nanomaterial's based adsorbent is highly recommended for dye pollutant removal[14,15]. The suitable figures[15] of merit in multi components dye system removing are based on development[15] of novel[16] methods which permits[15] its accurate simultaneous[15,16] determination of mixture. The encounters difficulty is serious peak overlapping[16] that subsequently[17] impossible for its direct determination in the mixtures using general equations like Beer–Lamberts. Derivatives spectroscopies efficiently are applicable[17] for resolving absorption peak overlaps through its separations and corrections of background interference[16]. These methods are based on searching[18] the wavelength which able to accurate[18] and repeatable[17] monitoring for each specie in the complexes without no interferences from the other targets compounds[16,17].The classical optimization of protocols failed for giving useful information of interactions between several variables[17].Central composites design or CCD[18] under surface methodologies can be applied[18] for handling of both variable involving and response without suffers above-mentioned[19] draw backs. These approaches enable us for making a suitable predicative models for applying several factor even in the presence[19] of complexes interaction for describing and finding the best optimized condition with less times elapsed[18,19].

In this Paper, MWCNT has been modified with N-(3-nitro-benzylidene)- N-tri methoxy silylpropyl-ethane1, 2diamine as the best adsorbents using FTIR and SEM. This adsorbent was used for the ultrasound-assisted removal of Thymol Blue and Erythrosine-B from aqueous solutions. The influences of variables initial Thymol Blue and Erythrosine-B as well as its interaction were investigated under response surface methodology[19].

EXPERIMENTAL

Materials and instruments

Thymol Blue and Erythrosine-B supply from Merck in Germany. The stocks solution[20] of one hundred mg/l for every dyes were getting dissolutions of ten milligram of which in 100 milliliter double distilled waters, separately and are suitable dilute[20] on performing for obtain worked solution with desired concentration[21]. R, N-(3-(tri meth oxysilyl)-propyl) ethylene diamine (TSPED) and 2-nitrobenzaldehyde (NBA) was purchased through SigmaAldrich. Multi-walled carbon nanotube was supplied via RiedelDeHean and other reagents[22] was used for analytical grades in the Merck Company[22]. The pH/Ion-meter was used for the pH measurement. Dye Thymol Blue and Erythrosine-B concentration was determined using Jasco UVVis spectrophotometric model V530, at wave-length of 430 nm and 580 nm, respectively[23].

The ultrasonic[24] bath including heating systems were used at frequencies 60Hz and power 130W. The morphologies of the NBATSPEDMWCNT were studied by SEM Hitachi, model S4160. The FTIR spectrophotometer for compound was recorded on the JASCO680" instrument in areaof 380-4200 cm^{-1} using potassium bromide pellets with molratio

1: 100 for the sample of potassium bromide the software MINITAB version 6.2.4.4 were used for experiment designing and its subsequent regression analysis[24].

Preparation of NBATSPED-MWCNT

At first step, tri methoxy silylpro pylethylene diamines support on MWCNT-(NH2-MWCNT) were synthesized through the reactions of 1.85 ml N-(3-(tri methoxysilyl)-propyl) ethylene diamine and 0.15 gramm MWCNT in 20 milliliter of dichloromethan under reflux at forty degree in the oil bath for twenty four hours. Then, the obtained solids were filtered. The products were filtered, washed with fifty milliliters of C2H5OH, double distilled water and then dried in oven for 10 h 50 ^{oC}. These step are presented in the Fig.1.

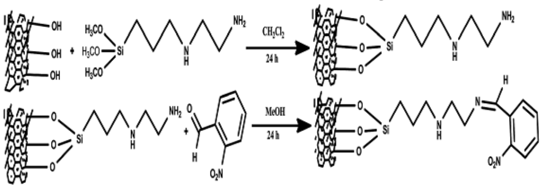

Fig.1. synthesis adsorb of NBATSPED-MWCNT

Ultra-sounded-assist a-multicomponent adsorb of Thymol Blue (TB) and Erythrosine-B (EB) on to NBATSPED-MWCNT

The batches process using NBA..MWCNT in presence of ultrasounds were applied for binaries adsorption of Thymol Blue and Erythrosine-B. The vessels were immersed in the ultrasonic baths for four minutes at room temperature and then the solution was centrifuged.

Adsorption equilibrium study

The equation and parameter of the isotherm of the research has been reported in Tables 1 and 2, respectively[20-26].

Desirability function (DF)

Desir abilities functions create the function of every individual responses leading to the final outputs of the global functions (D), max values of which support the achievements for optimum value[30]. The principles and applications of desirability functions for the best predications of real behaviors of adsorptions system were pointed out previously[30]. The desirability's profile indicates the predicted level of variable, which produce the most desirable responses.

Table. 1: Equation model used in this study

Linear equation	Model
$\log q_e = \log K_F + \frac{1}{n}\log X_e$	Freundlich isotherm
$\frac{1}{q_e} = \frac{1}{K_L q_m}(\frac{1}{X_e}) + \frac{1}{q_m}$	Langmuir isotherm

Table. 2: parameter applied in this work

Coefficients	Descriptions
K_F	Freundlich isotherm constant (dimensionless)
n	Freundlich isotherm exponent (dimensionless)
K_L	Langmuir constants
q_m	Maximum adsorption capacities

Table. 3 : Confirmation of the adsorptions for a dye at specific homogeneousness sites as the monolayers for NBATSPED-MWCNT surfaces

Run	X1	X2	X3	X4	X5	R $\%_{TB}$	R $\%_{EB}$
1	10	20	7	0.035	2	98	100
2	15	15	4	0.025	4	95	95
3	20	20	7	0.035	6	97.9	99.4
4	25	15	6	0.025	4	98	100
5	15	15	6	0.025	4	94.45	94.87
6	10	10	7	0.015	2	88	88
7	10	10	7	0.035	6	100	100
8	15	15	8	0.025	4	97.5	95.77
9	15	15	6	0.025	4	95	95
10	15	15	6	0.025	4	94.7	95
11	20	10	7	0.015	6	95	95
12	10	10	5	0.035	2	100	100
13	15	15	6	0.025	4	95	95
14	15	5	6	0.025	4	97	99
15	10	20	5	0.035	6	100	100
16	15	15	6	0.025	4	95	95
17	20	20	5	0.015	6	73	81.8
18	15	15	6	0.005	4	70	78.8
19	20	10	5	0.015	2	80	96.47
20	20	10	7	0.035	2	99.69	100
21	10	20	5	0.015	2	88.5	90
22	20	20	5	0.035	2	100	98.45
23	15	25	6	0.025	4	95	95
24	15	15	6	0.045	4	100	100
25	15	15	6	0.025	8	99.48	98.52
26	10	10	5	0.015	6	99.33	100
27	5	15	6	0.025	4	100	100
28	10	20	7	0.015	6	100	96
29	15	15	6	0.025	4	94.57	94.7
30	20	20	7	0.015	2	80	81.7
31	20	10	5	0.035	6	100	100
32	15	15	6	0.025	4	95	95

RESULTS AND DISCUSSTON

Based on theoretical calculation for comparison to experimental data the references have been considered[39-61].And based the experiments results and approaches of related works this work has been investigated[62-116].

Characterization of adsorbents

The FT-IR spectrums of NBASPEDMWCN (Fig. 2b), exhibits absorption peaks at 1715 cm^{-1} correspond to the stretching modes of vibrations for carbonyl group. The broad peak at 1086 cm^{-1} could assign to carbon oxygen stretching from phenolic, alcohlic, etherics group. These peaks can be assigned to the OH groups of moistures and carboxylic groups[34].The aromatic[35] C=C stretching are observed at 1459 cm^{-1}.The FT-IR spectrums (Fig. 3b.) displays the new peaks as a weak shoulders at 2931 cm^{-1}, which correspond to a stretching modes vibration for C–H bond in propyl[35] groups.

Statistical analysis

Central composites are designed as the most applicable types of RSM were applies for modeling and optimizing of concentrations for TB (X_1) and EB (X_2),pH(X_3),amounts of adsorbent (X4) and (X5) on the ultrasonic-assisted[36] adsorption of TB[37] and EB[38] dye through NBATSPED-MWCNT[38]. 5 independent variable was set at five level at which the R% of TB and EB dye as response were determined and exhibit in the Tables four and five. Analysis of the variance or ANOVA[38] were performed for evaluating the important[35] and effectives terms for modeling of the response based on F-test and p-values[36-38].

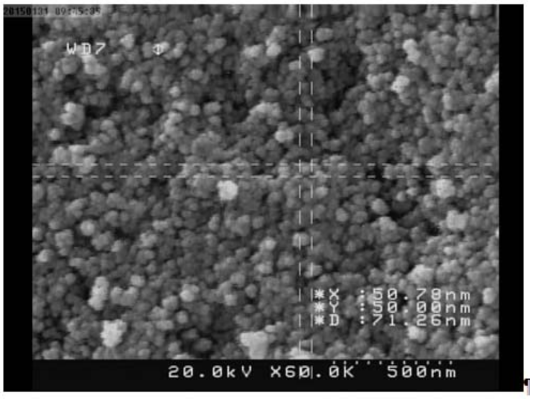

Fig. 2a. SEM images from preparation NBATSPED-MWCNT

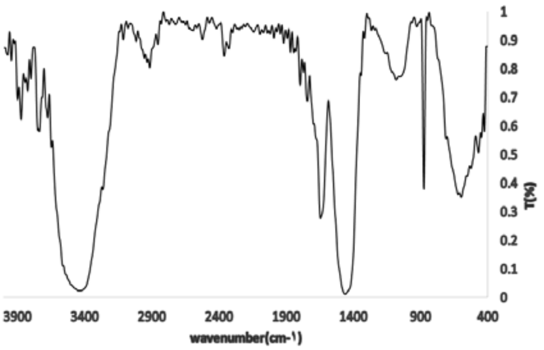

Fig. 2b. FT-IR transmittance spectrophotometer for preparation NBATSPED-MWCNT

Analysis of central composite designing

ANOVA was performed for obtaining information of the most important variable and its possible interaction (Table 5). Generally, they applied for the model of 11.16 and 8.48 of TB and EB respectively. The value calculated of the determination coefficient are R^2 0.96 and 0.94 for TB and EB respectively. The removal percentage of for TB and EB has been calculated. Therefore, the following semi-empirical expression applies for models of the removal percentage (R%) for TB and EB respectively[36].

For making other assessments on the applicability[37] of the model, the predicted and observed value of the removal percentages of TB and EB dye on NBATSPED-MWCNT were compared in (Fig. 3).

$$R\%=95.709-2.1767X_1-1.0258X_2+0.94833X_3+6.3233X_4+2.0955X_5-14.350X_1X_2+1.3388X_1X_3+2.9638X_1X_4-1.1638X_1X_5+0.69000X_2X_3+1.0650X_2X_4-1.3900X_2X_5-1.6612X_3X_4+1.4612X_3X_5-1.9138X_4X_5+0.66726X_1^2+0.91726X_2^2-0.020244X_3^2-2.8327X_4^2-0.41595X_5^2 \quad (5)$$

$$R\%_{EB}=95.775-0.88250X_1-1.3383X_2-0.21167X_3+4.6367X_4+12054X_5-1.7575X_1X_2+0.33625X_1X_3+1.0550X_1X_4-1.1513X_1X_5+1.2700X_2X_3+1.7388X_2X_4-0.21750X_2X_5+0.53250X_3X_4+1.4887X_3X_5-0.98000X_4X_5+0.94960X_1^2+0.94960X_2^2-0.20415X_3^2-1.7004X_4^2-0.12982X_5^2 \quad (6)$$

Response surface methodologies

Fig (3a, b) exhibits the removal percentages change versus the adsorbents dosages. The positive increasing in the dyes removal percentages with

Table. 6: Several isotherms including the correlation coefficient calculation dyes adsorption over NBATSPED-MWCNT

Isotherm	Equation	Parameters	Value of Parameters for TB(0.025g)	Value of Parameters For EB(0.025g)
Langmuir	$q_e = q_m\ bCe/(1+bC_e)$	Q_m(mg g-1)	50	100
		K_a (L mg-1)	1.53	0.487
		R2	0.997	0.998
Freundlich	$\ln q_e = \ln K_F + (1/n) \ln C_e$	1/n	0.24	0.55
		K_F (L mg-1)	3.66	4.09
		R^2	0.985	0.982

increasing in the adsorbent masses can be seen. Significant[38] diminishes in removal percentages at lower amounts of NBTSPEDMWCN are at attributes to higher ratio of dyes molecules. Fig. (3c) exhibits sonication times can be concluded the maximum adsorption of EB[37] could be riches when the sonication times were increased. Fig (3d) exhibits the removal percentages changing *versus* the adsorbent dosages.

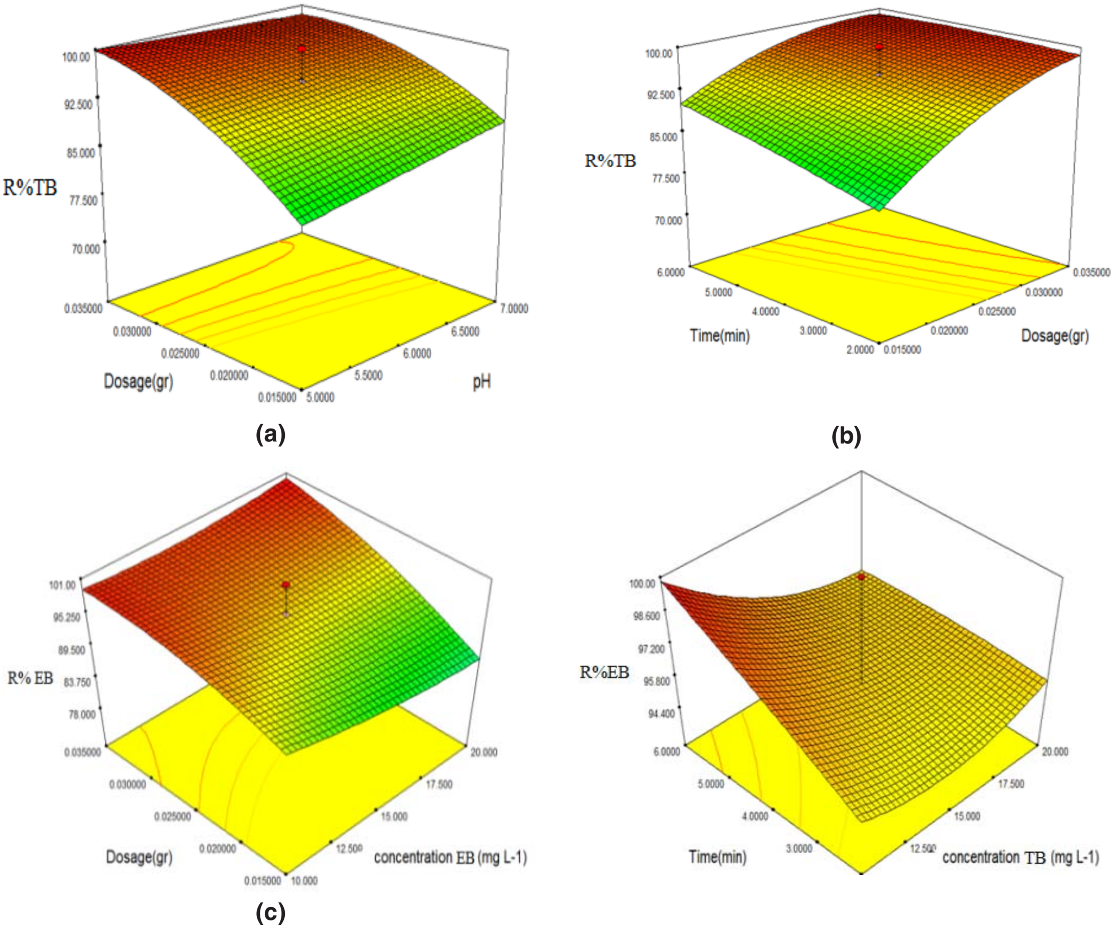

Fig. 3. surface of several dyes removing: (a) pH- adsorb dosages, (b) Time- adsorb dosages, (c) Time-concentrations of TB, Dosage-concentrations EB

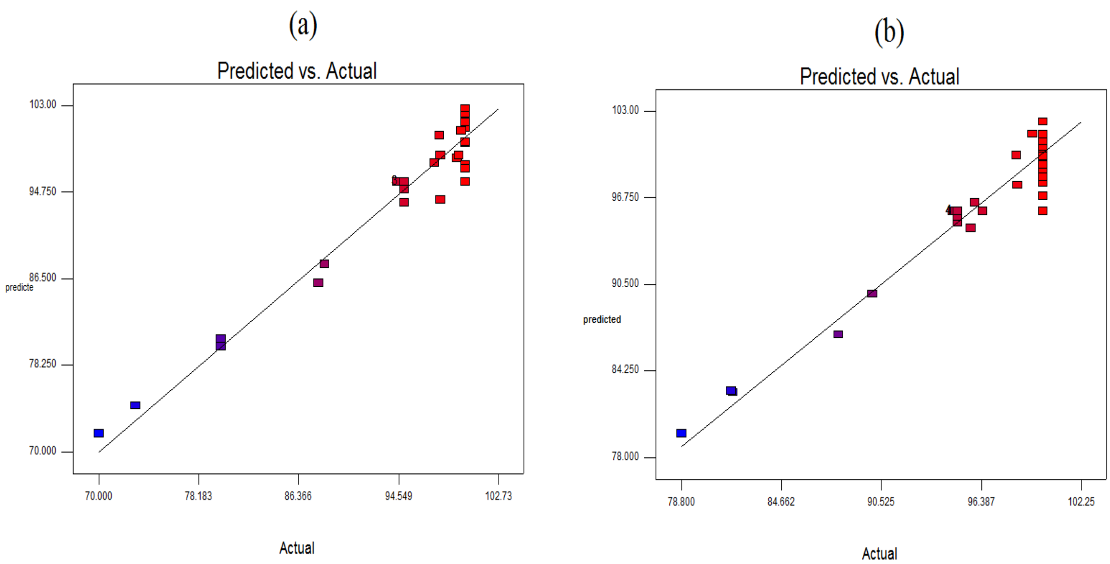

Fig. 4. (a) and (b) datum versus predication of datum for removing TB and EB.

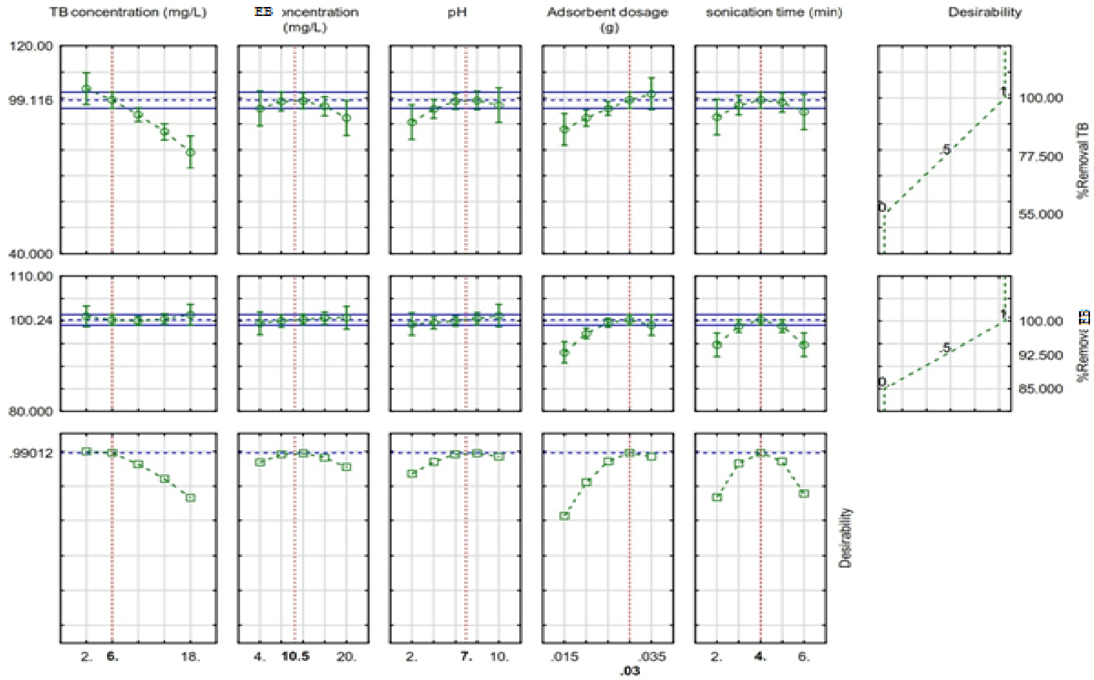

Fig. 5. Profiles for predication data and desirability functions for removing percentages of TB and EB.

CONCLUSION

Sonication's methods are effectives, very fast and sensitive to adsorbed materials compare to other methods especially, for the removal of Thymol Blue and Erythrosine-B.The influence of experiments parameter on the dye removal percentage was accomplished through experimental designing and the methodology of RSM.The adsorptions characteristics were examined with the variation of pH, sonication times, NBATPED-MWCNT dosages, initial TB concentrations and initial EB concentrations. The removal of TB and EB forms aqueous solution in short time 5 min. are feasible with high removal percentages at optimum pH (6.5) which are neutral and are advantages for the adsorption process.

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