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Research Article

Treatment of textile effluent by using Burflower tree (*Neolamarckia cadamba*) leaf powder: Batch biosorption study

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Abstract
The textile industry is one of the leading causes of major environmental problems worldwide. The textile dye effluents hold diverse kinds of dyes along with other contaminants. Various environmental legislations have been formulated and obligate the textile industries to treat the effluents prior to discharge into water bodies. The present study aimed to evaluate the efficacy of removing two dyes, Congo red and Methylene blue, present in raw textile effluent by employing the powdered leaves of the Burflower (*Neolamarckia cadamba*) tree as adsorbent. The experiment was performed in a laboratory scale. The surface characterization of the adsorbent was carried out using Brunauer-Emmett-Teller (BET) surface area analysis, CHN elemental analyzer, FTIR and SEM-EDX analyses. The effluent was characterised before and after the biosorption process to check the efficiency of the process on the various parameters of the effluent. The values of the examined parameters were found to be decreased after the adsorption process. The removal percentages of the two dyes (Congo red = 76.35% and Methylene blue = 85.8%) using the adsorbent were also estimated by batch experiment studies. The findings of this study infer that the adsorbent as mentioned above, can be used to treat dye effluents.

Keywords: Biosorption, Congo red, Effluent, Methylene blue, Removal efficiency

INTRODUCTION

The rapid industrial growth has led to various dreadful impacts on the environment. Out of the world's total annual production of synthetic dyes, textile industries consume several million tonnes, constituting around 56% (Sulyman *et al*.,2020) (Adegoke and Bello, 2015; Markandeya *et al.,* 2017). In the textile industries, a large volume of water is required during various operations, which generates enormous amounts of wastewater containing remarkable amounts of unfixed dyes (Markandeya *et al.,* 2017). Most of these industries dispose off their effluents into water bodies without treatment, thereby leading to environmental degradation. The indiscriminate discharge of industrial effluents without proper treatment poses a grave threat to environmental safety. Among all industrial sectors, the textile industries generate the most polluting wastewater in terms of effluent discharged volume and composition (Patel, 2018). The wastewater released from textile industries contributes towards the increase of Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Suspended Solids (SS), Dissolved solids, colour, heat and other soluble substances etc. (Howlader *et al*.,2022). The largest source of dye-containing effluents are textile, printing, food and cosmetics, which if discharged without proper treatment leads to serious environmental impacts (Mani and Bharagava, 2018). Such effluents contain toxic organic residues including a mixture of chemically versatile dyes leading to water (surface water as well as groundwater) pollution and soil pollution (Piaskowski *et al*.,2018). Most of these dyes are usually aromatic and heterocyclic compounds and very often recalcitrant, some of them are carcinogenic even. Out of the various structural varieties of dyes, viz., acid, reactive, basic, disperse, azo, etc., azo dyes are widely used by leather, textile, cosmetics and paper product industries

(Shah, 2014). The presence of azo dyes, which are found to be toxic, mutagenic and recalcitrant in textile wastewaters, can lead to various illnesses such as tumours, cancers and allergic reactions (Shah, 2018; Yaseen and Scholz, 2017). Because of their complex molecular structure and synthetic origin, the treatment of dyes is a bit difficult. Improper handling and insufficient treatment of spent effluents before disposal is the leading cause behind dye contamination. Even though many physical and chemical methods have been employed for the degradation of dyes, they have a few drawbacks, such as high running costs and high amount of sludge production (Rania Al-Tohamy *et al*.,2022). On the contrary, the biological methods are simple, cheap, produce lesser volumes of sludge, and are extensively used (Murungi and Sulaimon, 2022).

Over the last few years, the process of adsorption has gained paramount importance in industry and environment protection. Of late, scientists are paying substantial attention to the use of biological-based materials and their by-products as biosorbents for removing various contaminants owing to the presence of carboxyl, hydroxyl and amino groups over the surfaces of such materials (da Silva Alves *et al*.,2021). These groups are mainly responsible for the process of biosorption. Biosorption is a potential low-cost alternative method for the removal of dyes from textile effluent (Sardar *et al*.,2021). Biosorption takes advantage of the affinity of the dyes to adhere to surfaces for removing dyes using biomass (Vishnoi *et al*.,2020).

The decolorization of dyes using low-cost, naturally available products has gained much momentum in recent years. Applying natural biological adsorbents in industrial effluent treatment is emerging as an effective alternative technology to overcome the problems associated with physico-chemical methods. Burflower tree *(Neolamarckia cadamba)* is a large tropical tree with a broad crown and straight cylindrical bole having an average height of about 15 meters and belongs to the family Rubiaceae. Burflower tree is also known as Kadam tree. The leaves of this tree are broadly ovate, elliptic-oblong in shape with entire margin, pulvinus base, bitter in taste, mucronate apex, glabrous surface, pinnate venation and length varying from 7.5 to 18 cm and breadth 4.5 to 16 cm. Microscopic study of *N. cadamba* leaf powder showed the presence of unicellular, lignified trichomes, paracytic stomata, simple starch grains and sandy balls of calcium oxalate crystals. The qualitative chemical tests revealed the presence of saponins, steroids, alkaloids and carbohydrates in the leaf powder of *N. cadamba*. The leaves and bark of the plant have been reported to contain various medicinal properties such as astringent, anti-hepatotoxic (Kapil *et al*., 1995), antidiuretic, wound healing, antiseptic (Anonymous, The Wealth of India, 1992) and antihelminthic (Gunasekharan *et al*., 2006). Extracts of the leaves of *N. cadamba* possess the analgesic, antipyretic and anti-inflammatory activities. Keeping this fact in view, the present study attempted to investigate the biosorption potential of powdered leaves of Burflower (*Neolamarckia cadamba*) tree in removing Congo red and Methylene blue dyes from raw textile effluent.

MATERIALS AND METHODS

Reagents and chemicals

All chemicals used were of analytical reagent grade. All aqueous solutions were prepared in distilled water. The stock solution (1000 ppm) of both the dyes (Congo red and Methylene blue) were prepared by dissolving 1 g of the respective dye in 1000 ml distilled water. The reagents used for maintaining the pH were 1 N HCl and 1 N NaOH.

Preparation of the biosorbent

The leaves of Burflower (*N. cadamba*) tree were collected from the Gauhati University campus in Assam. Firstly, the leaves were washed 3-4 times with tap water followed by distilled water until the impurities were gone. The leaves were then dried in the shade for 24 hours and then dried in a hot air oven for 6 hours at 105°C. The leaves were then ground to fine powder, boiled to remove tannins and lignins in it, and dried again. Finally, the leaf powder was sieved to 100µ size. The adsorbent thus prepared was then stored in plastic bottles in a desiccator for future use (Ojha and Bulasara, 2015).

Sample collection

The textile dye effluent samples were collected from a local yarn dyeing plant located in the heart of Nalbari town, Assam, India. The sample was collected in 2 L polyethylene bottles. Standard procedures were followed during sampling and samples were transported to the laboratory and stored at 4°C.

Analysis of physicochemical parameters of the dye effluent

The physicochemical characterization of the effluent was made by analysing various parameters, viz., colour, temperature, pH, Electrical Conductivity (EC), Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Dissolved Solids (TDS), NO_3^-, SO_4^{2-}, PO_4^{3-}, turbidity, alkalinity and total hardness by the standard methods for examination of water and sewage as described in APHA (2023).

Batch experiments

The process parameters such as adsorbent dose, contact time and pH for the maximum removal of dyes were optimised. The 250 mL conical flasks containing 100 mL of effluent with a known biosorbent dose (0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 g) were agitated in a shaker at the desired speed. Blank solutions were run under the same conditions except for adding biosorbent. All the experiments were performed in replicates and at room temperature. After certain time intervals, the samples were taken out, filtered and centrifuged at 4000 rpm for 10 mins and the concentration of remaining dye solution was determined using UV-Vis spectrophotometer (Wen, C. Q., 2017). The percentage of dye removal from each sample was calculated by using the following relationship:

$$\% \text{ removal of dye} = \frac{(C_i - C_f)}{C_i} \times 100 \qquad \text{Eq. 1}$$

where, Ci = initial concentration of dye (mg/L) and C_f = final concentration of dye (mg/L).

Effect of adsorbent dose: The effect of adsorbent dose on the biosorption process was investigated by varying the mass of adsorbent (0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 g).

Effect of pH

The effect of pH was studied by changing the initial pH of the solutions from pH 2 to 10.

Effect of contact time

The effect of contact time was studied by agitating the solutions on the shaker for 10 to 90 minutes.

Decolorization of dye effluent

The effluent was diluted 1:10 for further experiments. The experiments were carried out in 250 ml Erlenmeyer flasks containing 100 ml of 1:10 diluted effluent at desired pH and concentration of biosorbent dosage. The flasks were kept under agitation using a shaker for the desired period of time. The effluent's residual concentration in dyes was determined after centrifugation at 4000 rpm. The effluent's hue change was determined by Spectrophotometric comparison of the sample with calibration curve prepared by measuring the absorbance of the known concentration of the dyes using a UV-visible Spectrophotometer. Similarly, blank without biosorbent was run as a control.

RESULTS AND DISCUSSION

Characterization of the adsorbent

The percentage contents of carbon, hydrogen and nitrogen analysed for the adsorbent are mentioned in Table 1.The relative adsorption performance of different adsorbent is highly dependent on the internal pore structure of each material (Manzoor *et al*.,2022). The surface area, pore size and pore volume of the adsorbent are mentioned in Table 2. The adsorbent's surface area was quite high, which implies that the adsorbent has a good adsorption capacity.

Neolamarckia cadamba primarily consists of indole alkaloids, terpenoids, sapogenins, saponins, terpenes, steroids, fats and reducing sugars (Batta and Rajput, 2021). Various phytochemical compounds have been identified from *N. cadamba* using phytochemistry approaches. The leaf extracts of *N. cadamba* revealed various secondary metabolites, including glycosides, alkaloids, tannins, phenolics, steroids, and flavonoids (Batta and Rajput, 2021). This tree possesses anti-diabetic, antioxidant, antimicrobial, anti-inflammatory effects. The presence of flavonoids in leaves of *N. cadamba* makes it effective in treating diabetes.

Fig 1. shows the FTIR spectrum of the fresh adsorbent. The broad absorption peak at 3325 cm^{-1} corresponds to the C-H and O-H stretching vibrations (Mosoarca *et al*.,2020). The peak at 2922 cm^{-1} indicates stretching of C-H bond of methyl and methylene groups (Uddin *et al*.,2009). The peak formed at 1724 cm^{-1} in the Fig.1 suggests the presence of C=O of carboxylic acid. The peak at 1608 cm^{-1} is due to C-C stretching vibrations. The presence of a secondary amine group is evident from the peak shown at 1519 cm^{-1}. The peak observed at 1446 cm^{-1} corresponds to the C=O group in carboxylate anion. The peak shown at 1255 cm^{-1} represents O–H wag ($-CH_2X$) of alkyl halides. The 1300 to 1000 cm-1 bands could be attributed to –C–O– linkages in alcohols, ethers, esters, carboxylic acids, etc.

The SEM micrograph (Plate 1) revealed the face texture and morphology of the fresh biosorbent. An observation of the SEM micrograph indicates the presence of many irregular and significant number of cavities/ pores on the surface of the biosorbent. The surface was highly heterogenous with many steps and burroughs and had a completely uneven topography. The presence of pores and internal surfaces are requisite for effective adsorbent (Manzoor *et al*.,2022).

The chemical characteristics of the surface of the adsorbent are given in Table 3. Energy-dispersive X-ray spectra (EDX) of the biosorbent (Plate 2) show the presence of carbon, nitrogen and oxygen on the surface. Indeed, KLP contains an elemental group of carbon, nitrogen and oxygen with atomic weight percentages of 69.11, 8.21 and 22.46, respectively. The low oxygen and high carbon contents are the major requisites for high adsorption capacity. The lower the oxygen contents, the higher the carbon contents of the samples under study, the more efficient the adsorbent.

Effect of adsorbent dose

The results (Fig.2a) indicated that with an increment in the adsorbent dose, the dye removal also increased and reached a maximum at 0.5 g and 0.4 g for Congo red and Methylene blue dyes, respectively. The increase in biosorption of dyes with an increase in adsorbent dose can be attributed to the fact that at higher

Table 1. CHN content of the adsorbent

Element	Carbon (C)	Hydrogen (H)	Nitrogen (N)
Percentage content	47.77	6.31	2.22

Table 2. BET surface area, pore size and pore volume of Burflower tree leaf powder

Surface area (m^2/g)	Pore size (Å)	Pore volume (cc/g)
113.455	16.019	0.058

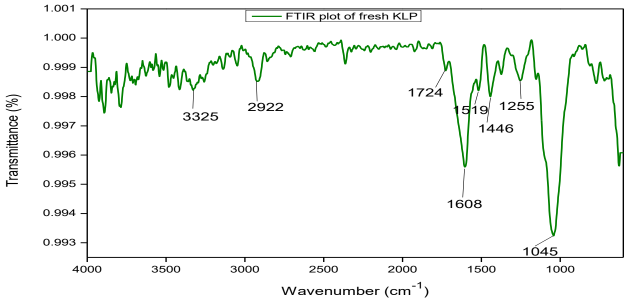

Fig. 1. *FTIR spectra of fresh biosorbent*

adsorbent doses, the surface area and, hence, the binding sites available for the attachment of dye molecules increase resulting in better biosorption (Mane and Babu, 2011). However, the dye removal decreases with a further increase in adsorbent dose. This might be due to the aggregation of biosorbent at higher doses, which leads to the blockage of binding sites on the surface of the biosorbent and hence, no further removal of dye molecules was achieved even at higher biosorbent doses (Mosoarca *et al.*,2020).

Effect of pH

The pH of the solution significantly affects the adsorption of dyes. The optimum adsorption capacity was achieved at pH 4 for both dyes (Fig.2c). The high removal capacities obtained at low pH values may result from the electrostatic attractions between the negatively

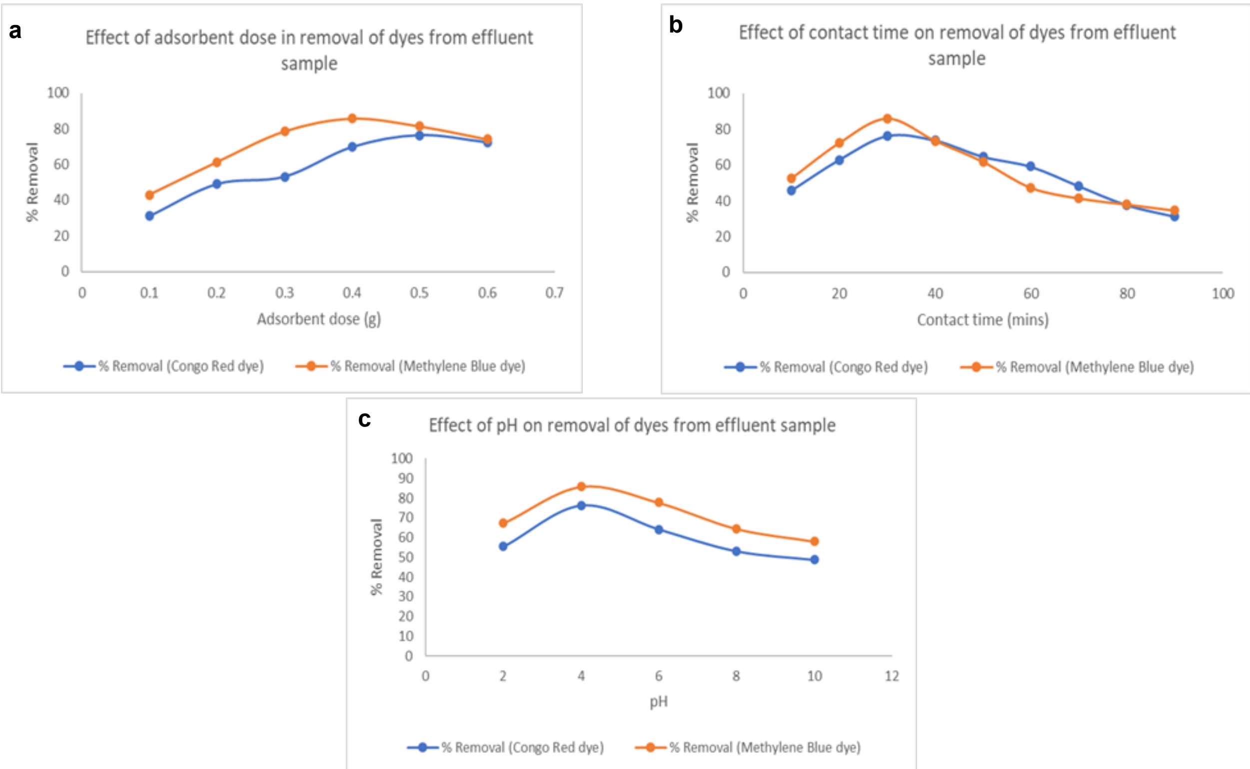

Fig. 2. *Effect of various parameters (a) Adsorbent dose, (b) Contact time and (c) pH on percentage removal of Congo red and Methylene blue dyes from the effluent sample*

Table 3. Elemental composition of fresh adsorbent

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
C K	61.51	69.11	3687.81	4.26	0.4607	1.0294	0.7275	1.0000
N K	8.52	8.21	89.04	15.58	0.0103	1.0023	0.1207	1.0000
O K	26.62	22.46	631.90	10.58	0.0527	0.9790	0.2023	1.0000
AuM	3.34	0.23	103.01	10.46	0.0252	0.5532	1.3669	0.9974

Table 4. Characterization of effluents before and after the biosorption process

Parameter	Before adsorption	After adsorption	Effluent discharge standards (inland surface water) as per CPCB
pH	10.2	8.3	5.5 to 9.0
EC (μScm^{-1})	2269	954	Not available
Biological Oxygen Demand (BOD)	378	45	30
Chemical Oxygen Demand (COD)	12750	239	250
Total dissolved solids (TDS)	4876	2365	2100
NO_3^- (mg/L)	9.49	8.17	10
SO_4^{2-} (mg/L)	41.65	36.3	Not available
PO_4^{3-} (mg/L)	15.67	11.2	5
Turbidity (NTU)	876	547	
Alkalinity	39	25	Not available
Total hardness (mg/L)	624	419	

charged dye anions and positively charged biosorbent surface. The decrease in the percentage removal of dyes with increasing initial pH values can be attributed to the change in surface characteristics and charge.

Effect of contact time

The percentage removal of dyes was observed (Fig.2b) to be increased initially with an increase in contact time and reached optimum at 30 minutes. But, with further increments in contact time beyond 30 minutes, there was no significant increase in the percentage removal of dyes.

Physicochemical parameters of the effluent before treatment

The dye effluent appeared dark purple, and the odour was quite unpleasant. The temperature of the sample was estimated to be about 22.8°C having a pH of 10.2. As seen in Table 4, the BOD and COD levels of the real textile effluent were found to be relatively higher than the standard limits. The BOD and COD levels were higher than the permissible limit, underlining the wastewater's oxygen-depletion properties. Thus, it is obvious that the wastewater was contaminated with organic load in addition to the dissolved and suspended matters.

pH is an important water quality parameter. Water having pH below 6.5 and above 8.5 is generally not recommended to be fit for consumption. The pH of the wastewater indicates that it is alkaline in nature. The alkaline pH of the water can cause severe physiological disturbances to most fish species of the water body, thereby inhibiting ammonia excretion leading to its accumulation and toxicity (Wen, C. Q., 2017). This implies that the discharge of such alkaline effluent into the water bodies without treatment can lead to serious aquatic pollution. The amount of total dissolved solids was also found to be quite high. The presence of high levels of TDS is due to the presence of inorganic matter in the effluent. Higher TDS content in water reduces it's utility for consumption, irrigation, and industrial purposes. Turbidity is a parameter to measure the status of polluted water because the higher grouping of dyes, ions and other contaminating agents will cause the wastewater

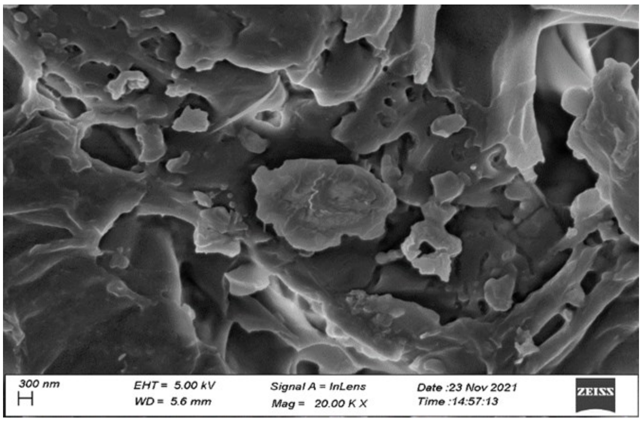

Plate 1. *SEM micrograph of fresh adsorbent*

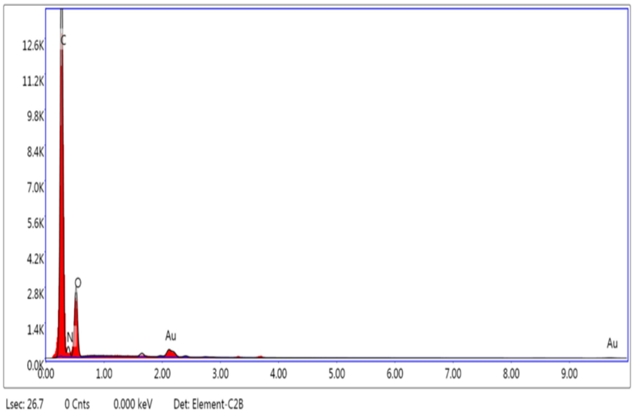

Plate 2. *EDX spectra of fresh adsorbent*

Table 5. Percentage removal of Congo red and Methylene blue dyes from industrial wastewater by using the adsorbent

Dyes	Initial concentration (mg/L)	Final concentration (mg/L)	Percentage removal (%)
Congo red	15.6	3.69	76.35
Methylene blue	20.35	2.89	85.8

to become polluted and turbid.

Reduction in the values of water quality parameters after treatment

After the adsorbent wastewater treatment, the BOD and COD levels were significantly reduced. The reduction in BOD and COD can be due to the adsorption of pollutants, mainly dyes, by the adsorbent. The characterization studies of the adsorbent revealed that the adsorbent has high porosity and high surface area, which resulted in effective capture of adsorbents from wastewater sample. Adsorbents with higher carbon content yield better performance in reducing COD of wastewater. The presence of hydroxyl groups and aromatic compounds in the adsorbent contribute towards physical adsorption (Wen, C. Q., 2017). In the present study, after adsorption, it was also evident that there was a reduction in the pH of the effluent. The turbidity value of the effluent decreased significantly after it was treated with the adsorbent. The reduction was due to the adsorption of pollutants by the adsorbent. Even the values of the rest of the parameters like TDS, alkalinity, total hardness and the ions like NO_3^-, SO_4^{2-}, PO_4^{3-} were seen to be declined following the adsorption using Burflower tree leaf powder.

The initial concentrations of the CR and MB dyes were calculated by using the calibration curves and the resultant final dye concentrations found are mentioned in Table 5. Based on this table, it was observed that after wastewater treatment, the percentages of dye removal were found to be 76.35 and 85.8 for Congo red and Methylene blue dyes, respectively. The removal percentages suggest that the adsorbent efficiently removes dyes from wastewater and can be applied in the field.

Conclusion

The present study concluded that removing various dyes from textile wastewater by adsorption on powdered leaves of the Burflower tree can be useful for controlling water pollution due to dyes. The results observed a decline in the pH, EC, TDS, BOD, COD, etc. after the effluent treatment by biosorption process. The dyes' removal percentages (Congo red and Methylene blue) by the adsorbent were significant enough. This shows that the biosorption process using Burflower tree leaves is effective in the treatment of textile effluents, and the adsorbent has substantial potential in the removal of dyes as well as other pollutants from effluents. Thus, the study can be helpful in the recovery of these dyes from the dye-contaminated sites using biosorption techniques involving low-cost and easily available adsorbents.

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Conflict of interest

The authors declare that they have no conflict of interest.

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