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Removal of Nickel and Iron from Metal Injection Moulding Industry Effluent by Adsorbent Method: A Comparative Study

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ABSTRACT

In the present investigation, the efficiency of rice husk in removal of nickel and iron from metal injection moulding industry effluent has been investigated. Adsorption was carried out in a batch experiment to investigate the parameters such as initial metal ion concentration, adsorbent dose, pH and contact time, under constant shaking of 100 mL sample in a heavy rotatory shaking apparatus for 2 hours. Analysis of physico-chemical characteristics of effluent was also carried out. The results revealed that maximum adsorption capacity of rice husk was about 92.84% for nickel using 8 g and about 90.12% for iron using 9 g at pH 9. Due to its good uptake capacity, rice husk has proved to be an excellent low-cost adsorbent for removing nickel and iron from effluent. The result showed that the percentage removal of nickel was high compared to iron.

INTRODUCTION

Toxic industrial waste, runoff from agricultural waste and discharge of untreated domestic waste are the main sources of water pollution (Adegoke et al. 2014). Heavy metal pollution is a major issue due to its toxic effect even at low concentration. Nickel and iron are the heavy metals used in the manufacturing process in metal injection moulding industry. Nickel is a non-biodegradable toxic metal which causes chronic bronchitis, reduced lung function, cancer, nasal sinus, etc. Iron is the 2nd most important metal for the survival and growth of the living organisms on the earth. But it is very toxic to the cells when given endogenous. Therefore, it becomes imperative to remove toxic metals like nickel and iron before discharging industrial effluent into water bodies.

Adsorption process is one of the best water treatment technique among various available methods. Adsorbent removes different pollutants in an easy way and economical and eco-friendly in nature. Rice husk is used as an adsorbent to remove nickel and iron from metal injection moulding industry effluent. The composition of main organic compounds like cellulose, hemicellulose and lignin renders rice husk as a possible adsorbent. In the present study, the applicability of rice husk for the removal of nickel and iron from industry waste water and effects of various parameters such as initial concentration of effluent, adsorbent dosage and pH has been investigated.

Rice Husk

Rice is a strategic crop all over the world and every year large amount of rice husk is produced. Structurally, it consists of crude protein (3%), ash (including silica 17%), lignin (20%), hemicellulose (25%) and cellulose (35%) which renders it suitable for metallic cations fixation. It has been used in the removal of some of the metal ions (Ajmal et al. 2003, Bishnoi et al. 2004, Dadhlich et al. 2004).

MATERIALS AND METHODS

Rice husk was collected from a rice mill at Ripponpete, Shivamogga Dist., Karnataka State and used as adsorbent for the removal of nickel and iron from effluent. It was washed with distilled water and dried in an oven at about 60°C for 4h and again washed with acetone and NaOH (0.3M) to remove dirt and other contaminants, dried in an oven at about 60°C for 4h and crushed until powdered fine particles were obtained. The powdered sample was examined by XRD (X-Ray Diffraction), SEM and BET analysis to assess the purity of the expected phases and the degree of crystallization, i.e. size, composition and crystal structure. The effect of different parameters, adsorbent and pH on the adsorption was carried out. pH was adjusted by HCL and NaOH (Thakur et al. 2013).

Instrumentation

Characterization of rice husk powder was performed by X-Ray diffraction (Rigaku Diffractometer) using Cu-Kα radiation (105406A°) in a θ - 2θ configuration and the average size of powder was calculated using Debye Scherrer's formula. Scanning Electron Micrograph determines the morphology of rice husk. Specific Surface Area (SSA) of rice husk was measured at 77 K by Brunauer-Emmett-Teller (BET) nitrogen adsorption-desorption (NOVA-1000 version 3.70 Instrument) (Morlu & Bareki 2017).

RESULTS AND DISCUSSION

Physico-chemical Properties of the Effluent

The physico-chemical properties of the metal injection moulding industry effluent are mentioned in Table 1.

Characterization of Rice Husk

X- Ray diffraction (XRD)

Using Cu-Ka radiation (105406A°) in a θ - 2θ configuration characterization of rice husk was performed by powder X- Ray diffraction (Rigaku Diffractometer). The average crystalline size of examined powdered rice husk was found to be around 28 nm and it was calculated using Debye Scherrer's formula (Fig. 1).

$$D = (K\lambda/(b \cos q)) \quad \ldots(1)$$

Table 1: Characterization of metal injection moulding industry effluent.

S.NO	Parameter	Before treatment	After treatment Nickel/Iron
1	pH	1	7/7
2	Temperature (°C)	28.9	24.4/25.1
3	Biological Oxygen Demand (mg.L-1)	30	25/26
4	Chemical Oxygen Demand (mg.L-1)	1250	956/980

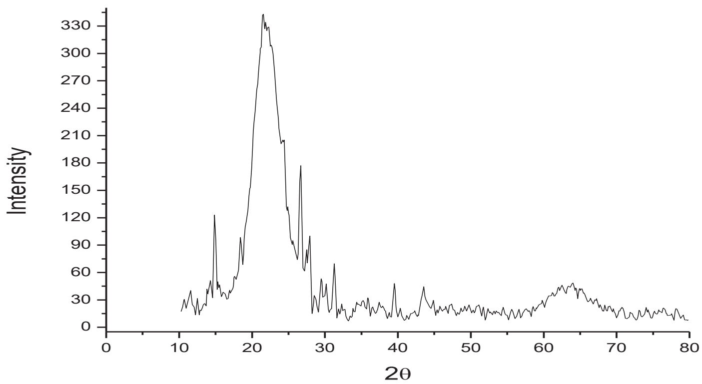

Fig. 1: XRD of the powdered rice husk.

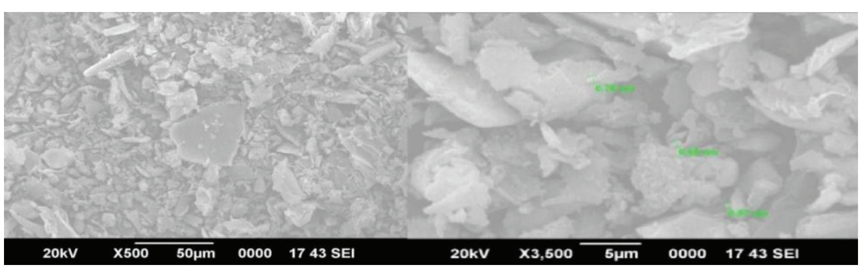

Fig. 2: Scanning Electron Micrograph of powdered rice husk.

Table 2: Surface properties of powdered rice husk.

	Surface area	Pore volume	Average pore diameter
Rice husk	13.386 m^2/g	0.022 cc/g	1.557 nm

Table 3: Concentration of nickel and iron before and after treatment with rice husk.

Concentration of Effluent	Nickel (mg/L)		Iron (mg/L)	
	Before	After	Before	After
25%	15.01 ± 0.005	6.93 ± 0.005	21.82 ± 0.005	12.02 ± 0.005
50%	18.02 ± 0.01	5.65 ± 0.01	24.31 ± 0.005	9.64 ± 0.01
75%	20.22 ± 0.005	10.97 ± 0.01	27.63 ± 0.005	16.59 ± 0.01
100%	23.14 ± 0.005	13.94 ± 0.01	30.47 ± 0.005	19.64 ± 0.01

Scanning Electron Microscope (SEM)

The SEM image of powdered rice husk is shown in Fig. 2 indicating the aggregation particles and cluster shape with sharp edge. The properties of the powdered rice husk are mentioned in Table 2

Bet Surface Analysis

To determine the adsorption efficiency of nickel and iron, batch experiments were conducted and the effect of parameters such as adsorbent dose, pH and contact time was studied.

A known weight of rice husk with 100 mL effluent of different dilutions was kept for treatment in heavy rotatory shaking apparatus for 2 hours. Initial concentration of nickel and iron was determined and found to be as follows. Nickel, 15.01 mg/L in 25% concentration, 18.02 mg/L in 50% concentration, 20.22 mg/L in 75% concentration and 23.14 mg/L in raw effluent (100% concentration). After treatment samples were filtered using Whatman No. 41 filter paper and measured by AAS. The concentration of nickel reduced to 6.93 mg/L (25% concentration), 5.65 mg/L (50% concentration), 10.97 mg/L (75% concentration) and 13.94 mg/L (raw effluent) (Table 3). Maximum reduction of 18.02 mg/L (68.64%) was observed in 50% concentration (Fig. 3) i.e. from 18.02 ± 0.01 to 5.65 ± 0.01 (Table 3).

Initial concentration of iron was 21.82 mg/L in 25% effluent concentration, 24.31 mg/L in 50% concentration, 27.63 mg/L in 75% concentration and 30.47 mg/L in raw effluent. After treatment the concentration of iron reduced to 12.02 mg/L (25% concentration), 9.64 mg/L (50% concentration), 16.59 mg/L (75% concentration) and 19.64 mg/L in raw effluent (Table 3). Maximum reduction of iron was found to be 24.31 mg/L (60.34%) in 50% concentration (Fig. 4) i.e. 24.31 ± 0.005 to 9.64 ± 0.01 (Table 3) because of availability of more adsorption activated sites. Metal ions are easily adsorbed on vacant sites at low concentration. The percent removal of nickel and iron was calculated by the formula:

$$\text{Percent removal} = \frac{C_o - C_i}{C_o} \times 100 \qquad \ldots(2)$$

Where, C_o is initial concentration and C_i is final concentration of nickel/iron metal. Further experiment was carried out based on the results obtained.

To know the effect of adsorbent, experiment was carried out with different dosage of rice husk from 1-10g/100 mL at pH 7 in conical flasks in a heavy rotatory shaking apparatus for 2 hours. Maximum percentage removal of nickel was 92.84% at 8g/100 mL (Fig. 5) and iron was 90.12% at 9g/100 mL in 50% concentration (Fig. 6).

Nickel reduced from 18.02 ± 0.01 to 1.29 ± 0.01 andiron decreased from 24.31 ± 0.005 to 2.40 ± 0.005 in 50% concentration (Table 4). It was observed that the percentage removal of nickel and iron increased with increased adsorbent dose which means that when the concentration of adsorbent increases, the availability of high active sites will increase the adsorption capacity which helps to remove the metal ion.

In the adsorption process, pH is considered as the speciation and surface charge of the adsorbent because of its great impact in the metal ion removal. Experiment was carried out with a pH range of 1, 3, 5, 7, 9 and 11. The results show that nickel removal increased from 30.24% to 92.23% (Fig. 3) i.e. 18.02 ± 0.01 to 1.40 ± 0.005 (Table 5) and iron increased from 16.7% to 90.25% (Fig. 4) i.e. 24.31 ± 0.005 to 1.93 ± 0.01 in 50% effluent concentration. Maximum removal of nickel was at pH 9 with 8 g of rice husk and it slightly decreased at pH 11 (Fig. 7). Maximum removal of iron was at pH 9 with 9 g of rice husk and it gradually decreased at pH 11 (Fig. 8). The results easily show that the removal of nickel and iron by rice husk increases with increasing pH and culminates at pH 9.

Similar work has been reported by Abbas et al. (2013) on utilization of Iraqi rice husk in the removal of heavy metals from wastewater. The results showed that maximum removal of Ni by Iraqi rice husk was 95.82% and the percentage removal decreased with increasing pH respectively. Hegazi (2013) worked on removal of heavy metals from wastewater using agricultural and industrial wastes as adsorbents. The result indicated that the maximum removal of iron was 99.25%.

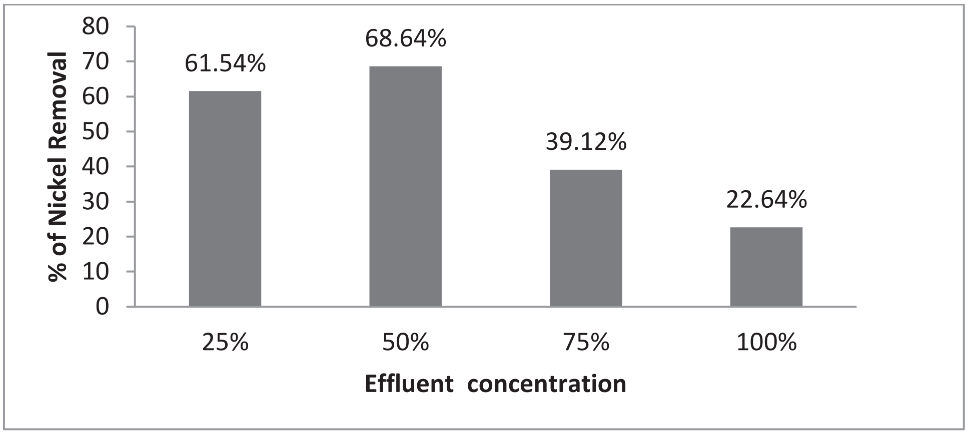

Fig. 3: Removal of nickel with rice husk at different effluent concentration.

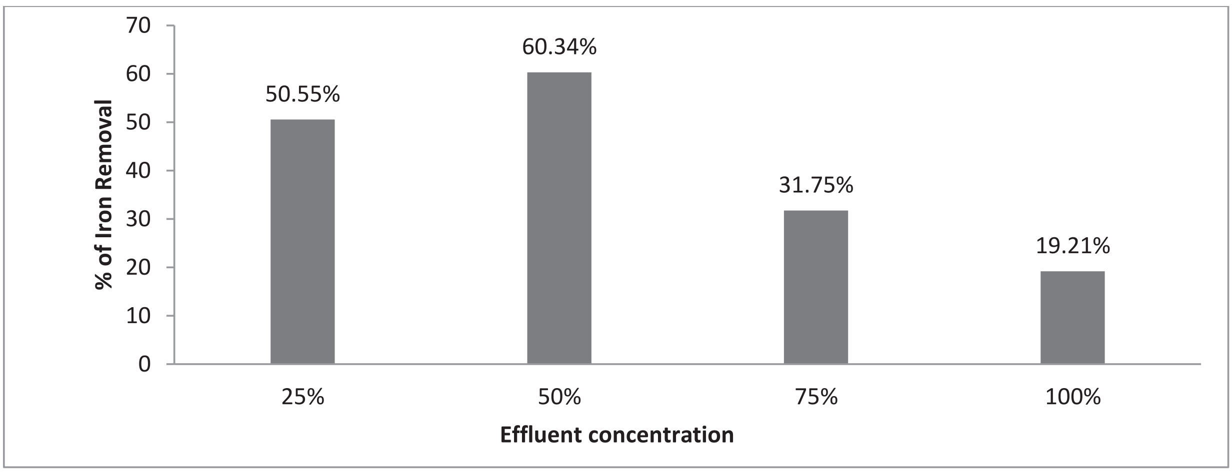

Fig. 4: Removal of iron with rice husk at different effluent concentration.

Table 4: Effect of adsorbent on nickel and iron before and after treatment (1-10g/100 mL at pH 7).

Adsorbent dosage (g)	Nickel (mg/L)		Iron (mg/L)	
	Before	After	Before	After
1	18.02 ± 0.01	9.91 ± 0.02	24.31 ± 0.005	18.73 ± 0.005
2	18.02 ± 0.01	8.97 ± 0.005	24.31 ± 0.005	16.93 ± 0.005
3	18.02 ± 0.01	7.80 ± 0.005	24.31 ± 0.005	13.44 ± 0.01
4	18.02 ± 0.01	6.97 ± 0.01	24.31 ± 0.005	11.65 ± 0.005
5	18.02 ± 0.01	5.65 ± 0.005	24.31 ± 0.005	9.62 ± 0.05
6	18.02 ± 0.01	4.41 ± 0.01	24.31 ± 0.005	7.88 ± 0.005
7	18.02 ± 0.01	2.97 ± 0.01	24.31 ± 0.005	5.94 ± 0.01
8	18.02 ± 0.01	1.29 ± 0.01	24.31 ± 0.005	4.12 ± 0.005
9	18.02 ± 0.01	1.52 ± 0.10	24.31 ± 0.005	2.40 ± 0.005
10	18.02 ± 0.01	1.56 ± 0.005	24.31 ± 0.005	2.50 ± 0.005

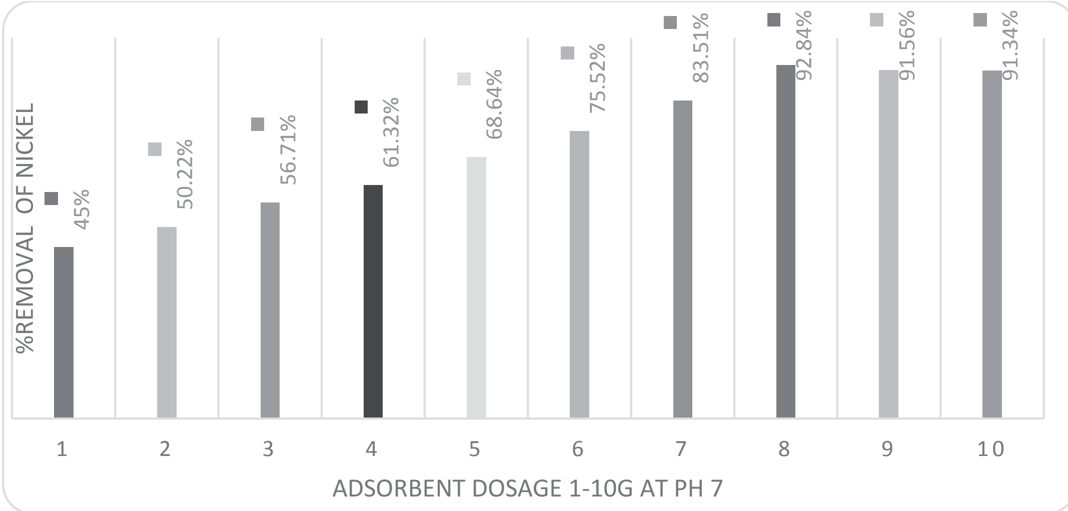

Fig. 5: Removal of nickel for different adsorbent dosage (1-10g/100 mL at pH 7).

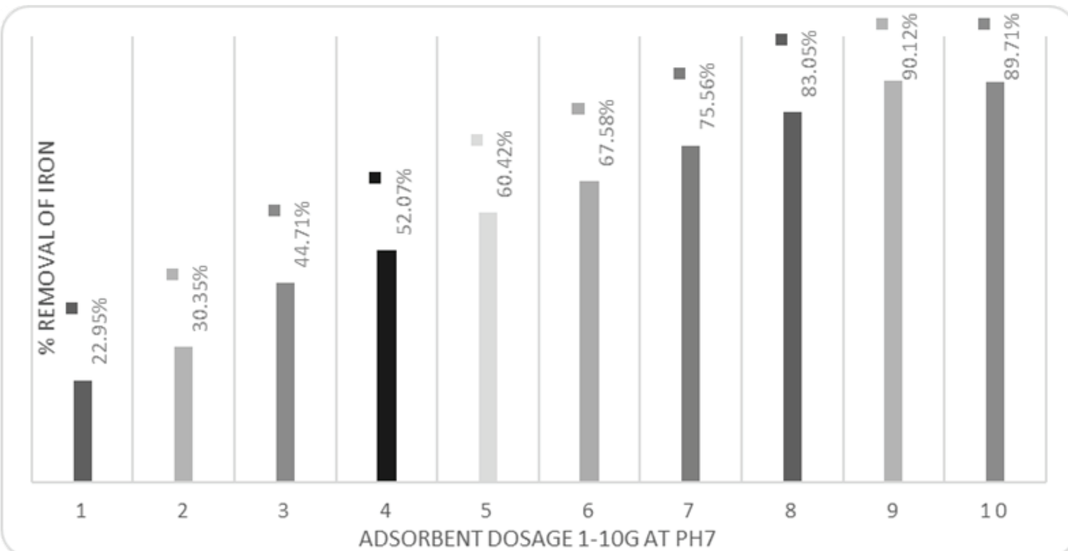

Fig. 6: Removal of iron for different adsorbent dosage (1-10g/100 mL at pH 7).

Table 5: Effect of pH on the adsorption of nickel and iron before and after treatment with rice husk.

pH range	Nickel (mg/L)		Iron (mg/L)	
	Before	After	Before	After
1	18.02 ± 0.01	12.57 ± 0.01	24.31 ± 0.005	20.25 ± 0.01
3	18.02 ± 0.01	8.20 ± 0.005	24.31 ± 0.005	12.63 ± 0.005
5	18.02 ± 0.01	5.07 ± 0.005	24.31 ± 0.005	9.12 ± 0.005
7	18.02 ± 0.01	2.93 ± 0.005	24.31 ± 0.005	2.37 ± 0.01
9	18.02 ± 0.01	1.40 ± 0.005	24.31 ± 0.005	1.93 ± 0.01
11	18.02 ± 0.01	1.97 ± 0.005	24.31 ±0.005	2.02 ± 0.01

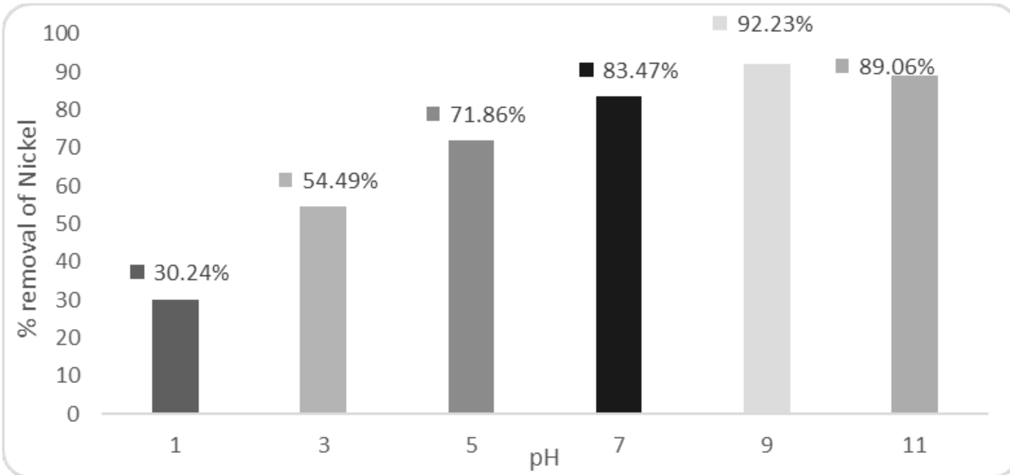

Fig. 7: Effect of pH on the adsorption of nickel.

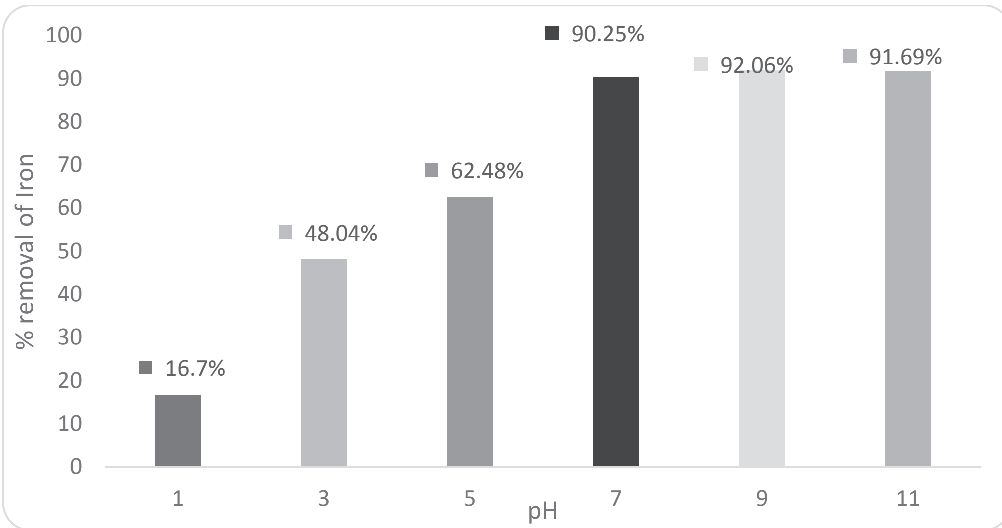

Fig. 8: Effect of pH on the adsorption of iron.

CONCLUSION

The adsorption efficiency of rice husk and comparative study of nickel and iron removal has been studied. XRD and SEM analysis showed that the average crystalline size of powdered rice husk was around 28 nm and morphologically was cluster shape with sharp edge. BET analysis explained the surface properties like pore diameter, pore volume and surface area of powdered rice husk. It was observed that 92.84% nickel was removed by 8 g of rice husk and 90.12% iron was removed with 9 g of rice husk in 50% concentration with 2-hour treatment at 25°C. The maximum removal of nickel and iron was obtained at pH 9. Comparatively rice husk was found to be efficient in removal of nickel than iron.

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