

Treatment of Dairy Wastewater with Pretreatment Techniques and Sequencing Batch Reactor for the Removal of Pollutants: Kinetics and Optimization

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In order to remove pollutants like BOD, COD, TKN, TP, TSS, sulphates, chlorides, oil and grease effectively, this study outlines the treatment of dairy effluent by employing the advanced oxidation process (AOP) as pretreatment method with a sequencing batch reactor. It also describes the impact of aeration time and bacterial mass concentration (MLSS) in the sequencing batch reactor for pollutant removal. The high percentage of nutrient contents can be removed by the sequencing batch reactor (SBR), which only needs a single tank for the entire treatment procedure. The pretreatment technique of a combination of ozone and AOP was introduced into the dairy effluent to increase the removal efficiency of pollutants. In this research, the optimal conditions for ozone were a pH of 10, a reaction time of 5 minutes, and an amount of H_2O_2 of 10 mL/L. The experiments were conducted in the 4 L Plexiglas reactor and the analysis was carried out for different aeration times of 4, 6, 8, 12, 16 and 24 h at the different MLSS concentrations of 2100, 2400, 2900, 3200, 3700 and 4200 mg/L after the addition of ozone and hydrogen peroxide. In this analysis, the higher percent removal of BOD, COD, TKN, TP, TSS, sulphates, chlorides, oil and grease was obtained for the MLSS of 2100 mg/L at the aeration time of 16 h. The percent removal of above pollutants for the above MLSS concentration was in the order of 99.8%, 99.6%, 76.7%, 99%, 99.3%, 34%, 46% and BDL, respectively. The GC-MS analysis was carried out to find the level of contaminants present in the raw and treated water. Further, the sludge analysis was done for the wasted sludge to find its characteristics and its uses. This study suggests that at low MLSS concentrations, a large percentage of pollutants can be removed.

Keywords: Advance oxidation process, Sequencing batch reactor, Aeration time, Ozone, Hydrogen peroxide, Plexiglas reactor.

INTRODUCTION

Milk is considered one of the main commodities entering into everyone's life. This should be of a high grade, free of any potentially harmful bacteria [1]. The wastewater discharge from the dairy industry is mainly composed of organic matter and suspended solids [2,3]. Carbohydrates, proteins and fats are the main contributors of organic load to these effluents [4]. The wastewater from the food industry is always having high nutrient content, especially in nitrogen [5,6]. Some of the operations performed in the dairy industry are processing of raw materials, packaging and storing of the finished products [1]. The use of acid and alkaline cleaners in the dairy industry influences the type of water [4].

In terms of industrial wastes, those from the dairy sector are among the most polluting. About 2.5 L of wastewater is

generated for the processing of 1 L of milk [1]. An increase in industrialization and urbanization leads to an increase in the rate of water pollution. So, the environmental protection agencies imposed regulatory prohibitions to control the pollution [7]. The conversion of 100 kg of milk into cheese yields only 10-20 kg of cheese, resulting in 80-90 kg of whey, which further exacerbates the environmental issues [8]. The main environmental problem related to dairy waste is air pollution, water pollution and loss of biodiversity. Thus, the effluent has to be treated well before discharging this effluent into the water bodies [3].

Ozonation is used to disinfect the water for drinking purposes and has many advantages in the food industry too [9]. The use of AOP for the treatment of industrial wastewater is scarce [10]. Ozone alone can't able to degrade organic compounds like aliphatic carbon, amides and nitroso compounds. For that

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hydrogen peroxide is added to degrade the above organic compounds [11]. The combination of ozone and hydrogen peroxide decomposes the organic matter present in the effluent through hydroxyl radicals [8,11]. The conventional treatment methods like activated sludge process (ASP), fluidized bed reactors (FBR), trickling filter and upflow anaerobic sludge blanket (UASB) doesn't remove all organic compounds and nutrients. It can be overcome by SBR [12]. The main advantage of SBR technology is the process of aerobic and anaerobic can be done in one tank. The SBR systems can reduce the nitrogen content by nitrification and denitrification [6]. It is done by five processes where the effluent is filled by gravity or by pump followed by aeration, settle, decant and idle [12]. The biosludges are further recycled and can be used as fertilizer, building material and bio-fuel [6].

The working principles of SBR are based on the activated sludge process [13]. The advantages of SBR are low cost and simplicity in operation, flexibility, less area requirement and can be used for a wide variety of wastewaters [6]. For instance, Omil et al. [14] reported that about 61.4% of nitrogen removal and increase the DO concentration, which will increase the removal efficiencies by using SBR technique. Sirianuntapiboon et al. [15] also suggested that the removal efficiencies of BOD, COD, TKN, TP and TSS are dependent on the aeration time and MLSS concentration. Meric et al. [16] studied that AOP removed 53% of COD which is high when compared to coagulation and flocculation. Moreover, Mohseni & Bazari [17] also achieved above 90% of COD removal efficiency by using SBR technique. Lin & Cheng [18] revealed that 93.6% of COD and 91.8% of BOD were removed in municipal wastewater by using SBR along with chemical coagulation. Interestingly Thakur et al. [19] suggested that biological treatment of wastewater is the most effective when compared to coagulation, adsorption and chemical oxidation techniques since biological methods are cost-effective and no secondary pollutants will be produced.

The high EC value is attributed to high salinity and high mineral content. The high value of chloride represents the use of a high amount of sanitizers, detergents and washing powder in the dairy industry [20]. Maranon et al. [21] reported that 69% of COD removal is achieved for the hydraulic residence time (HRT) of 58 h, while Fernandes et al. [22] reported that upto 70% of TSS removal efficiency can be achieved. The present work was carried out for treating dairy wastewater with pretreatment techniques and SBR. In the pretreatment study, the dairy effluent was treated with ozone and hydrogen peroxide. The SBR study was conducted by varying the aeration time (4, 6, 8, 12, 16 and 24 h) and MLSS concentration (2100, 2400, 2900, 3200, 3700 and 4200 mg/L). These studies are conducted to find the best removal percentage efficiencies of BOD, COD, TKN, TP, TSS, chlorides and sulphates. The effluent from SBR is passed to the sand filter and activated carbon bed to remove the suspended solids and colour of the effluent.

EXPERIMENTAL

Collection and preservation of dairy effluent: The dairy wastewater was collected from the local Ambattur dairy milk processing unit. The raw effluent flowed through the pipes where the sample was collected. The collected dark cream white samples were sealed and refrigerated properly at 4 °C.

Characterization of dairy wastewater: The collected effluent was analyzed for pH, BOD, COD, TKN, phosphates, TSS, sulphates, oil, grease and chlorides as per APHA [23]. The reagents required for BOD are phosphate buffer solution, magnesium sulphate solution, sodium sulfite solution and ammonium chloride solution. The analysis of BOD was done by calculating the initial and final depletion of dissolved oxygen for 5 days (Part 5210 of APHA) [24]. For COD, the test was carried out at 150 °C in a COD digester for 2 h. After titrating with FAS the sharp colour change will happen (Part 5220 of APHA) [25]. Total Kjeldahl Nitrogen (TKN) analysis was carried out by the digestion and distillation process. The digestion process was done at 450 °C for 3-4 h. Total suspended solids (TSS) was analyzed by using the gravimetric method (Part 2540 of APHA) [26], whereas chloride estimation was done by using the argentometric method (Part 4500 of APHA) [27]. Phosphates and sulphates were analyzed by using UV-Vis spectrophotometer (Part 4500 of APHA) [28].

Pretreatment techniques: The pretreatment technique consists of introducing the ozone through an ozone generator and the addition of hydrogen peroxide to the raw dairy effluent. This pretreatment work is required to miminize the pollutant load before introducing it into the SBR. It will lead to results in high removal efficiency in SBR. The ozone was produced at the rate of 15 g/h. According to the findings of the prior studies, the mixture of ozone and hydrogen peroxide was more effective than either component used alone [29].

The previous work indicated that the pH of 10 at ozone for 5 min produces the best results and 10 mL/L of hydrogen peroxide concentration produces better results than others [30]. The amount of COD in raw dairy effluent is decreased by 42% as a result of this study. To further reduce pollutant loads, this effluent was sent to the sequencing batch reactor, where a biological procedure would be carried out.

Design and experimental setup of sequencing batch reactor (SBR): The experimental setup consists of a raw sample tank, pretreatment tank, SBR, sludge collection tank and effluent collection tank. These tanks were made up of Plexiglas and the overall dimension of the SBR was $14 \text{ cm} \times 14 \text{ cm} \times 30$ cm. There was capacity of total of 6 L in the reactor, with only 4 L actually being used. The feed tank of 3 L capacity was used for feeding the effluent into the pretreatment tank. Feed was made to flow by gravity providing a difference in head between the feed tank and pretreatment tank. The volume of the pretreatment tank and the effluent collection tank was 3 L. The pretreatment tank was used to do the pretreatment techniques like the addition of ozone and hydrogen peroxide. This effluent was made to flow by gravity to the SBR. The SBR was provided with two openings for effluent collection and sludge collection. It also consists of air compressor and three diffusers for the supply of air to the reactor. The effluent and sludge were collected in separate tanks after treatment, with the effluent being collected in the effluent collection tank.

Preparation of seed sludge for biomass development: The thickened sludge from the dairy effluent treatment plant was taken to develop the biomass concentration in SBR. The brown sludge was taken from the bottom of the secondary clarifier unit. Then the sludge has to be settled for a day to get further thickened sludge. About 10% v/v of thickened sludge was added to the reactor. The concentration of biomass will be increased by adding the desired amount of cow dung and nutrients like urea and diammonium phosphate (DAP). MLSS was checked regularly to achieve the desired bacterial mass concentration.

Optimization of aeration time and MLSS: After the required MLSS achieved, the reactor was operated for different aeration times and different MLSS concentrations. The dairy effluent was added to the reactor where the biomass is present. Different sets of experiment were conducted by varying the MLSS concentration to 2100, 2400, 2900, 3200, 3700 and 4200 mg/L at various aeration times of 4, 6, 8, 12, 16 and 24 h for each biomass concentration. This type of work was carried out to analyze the percentage removal efficiency of BOD, COD, TP, TKN, TSS, chloride and sulphates. The first set of experiments was done at MLSS of 4200 mg/L for aeration times of 4, 6, 8, 12, 16 and 24 h. Next, the MLSS was brought down to 3900 mg/L by discarding the targeted volume of sediment, and aeration will be performed at various aeration rates. The fill time and settle time of the effluent were 10 and 30 min, respectively. Similarly, the other sets of experiments were done by wasting the sludge to MLSS of 3200, 2900, 2400 and 2100 mg/L at different aeration times. These sets of experiments were used to find the better MLSS and aeration times for maximum pollutant removal.

RESULTS AND DISCUSSION

Characteristics and emission load analysis of dairy wastewater: The collected wastewater sample from the dairy industry was examined for its initial properties, including pH, BOD, COD, TP, TKN, TSS, chloride, sulphate, oil, grease and electrical conductivity. The pH of the collected dairy effluent was found to be slightly acidic 5.57 and the temperature of the sample was 29.2 °C. The organic matters present in the dairy effluent are normally high. The BOD and COD of the analyzed dairy effluent were 1975 and 3727 mg/L. The higher the COD value, the more serious the pollution by organic matter by water. The other characteristic parameter values are given in Table-1. Milk, cheese, and curd crumbs were the primary sources of the suspended solids found in dairy effluent. The electrical conductivity of this effluent was 1.613 ms/ppt and also found that oil and grease were below the detectable limit.

Effect of MLSS at different aeration times: The dairy effluent from the sample storage tank has been sent to the pretreatment tank where all the pretreatment works like the addition of ozone, hydrogen peroxide was carried out. After the pretreatment work, the effluent was sent to the SBR where the bacterial mass concentration (MLSS) varied from 2100 to 4200 mg/L at different aeration times of 4, 6, 8, 12, 16 and 24 h. Following the completion of the aeration process, the wastewater

TABLE-1 CHARACTERISTICS OF DAIRY WASTEWATER SAMPLE								
Parameters Value								
pH	5.57 @ 29.2 °C							
BOD (mg/L)	1975.3							
COD (mg/L)	3727							
Total Kjeldahl nitrogen (mg/L)	26.56							
Total suspended solids (mg/L)	930.2							
Total phosphates (mg/L)	52.27							
Chlorides (mg/L)	240							
Sulphates (mg/L)	70							
Oil and grease (mg/L)	BDL							
Electrical conductivity (ms/ppt)	1.613							

was allowed to settle and then decanted. The decanted effluent was sand filtered followed by activated charcoal for adsorption. The treated effluent was analyzed for different parameters like BOD, COD, TKN, TP, TSS, chlorides, sulphates for finding the removal efficiency. The results are tabulated in Table-2.

Based on Fig. 1, it was found that increasing concentration and duration has a greater impact on these systems. However controlling the quantity and the quality of sludge has more effect in the removal of viable pollutants from the effluent. When the quantity of MLSS increases above a certain threshold, its effectiveness starts to decrease. From 2100 mg/L to 2900 mg/L an increase in the MLSS concentration increase in the removal of pollutants such as COD, BOD, solids and phosphorus, etc. However, further increase in MLSS concentration the removal efficiency got decreases because excessive sludge concentrations might affect the efficiency of oxygen transfer and delay the nitrification and de-nitrification process. Similarly, an increase in the aeration time beyond 6 h no significant changes were observed in the pollutants removal. Hence, the optimum level of 2900 mg/L of MLSS concentration and the aeration time of 6 h was maintained for additional studies.



Fig. 1. Effect of MLSS concentration and aeration time in COD removal

Adsorption by activated charcoal: TAfter optimising the MLSS concentration and aeration duration, the effluent was pumped through a sand bed filter to remove floating debris, and then the filtrate was passed through an activated carbon bed to remove any remaining stains. This effluent was analyzed for BOD, COD, TKN, TP, TSS, chlorides, sulphates, oil, and grease removal effectiveness before and after treatment.

TABLE-2 FOR DIFFERENTS MLSS (mg/L) AND AERATION TIMES												
Parameters	Aera	ation = 4 h	Aera time	ation = 6 h	Aera time	ation = 8 h	Aera time =	ation = 12 h	Aera time =	tion = 16 h	h Aerati	
	Conc.	R (%)	Conc.	R (%)	Conc.	R (%)	Conc.	R (%)	Conc.	R (%)	Conc.	R (%)
MLSS-2100 n	ng/L											
pН	7.95	-	8.19	-	8.56	-	8.94	-	8.98	-	8.88	-
EC	0.315	-	0.469	-	0.501	-	1.694	-	1.356	-	1.569	-
ROD	91.19 276.3	95 03	49.7 146.3	97	13.00	99	9.35	99	BDL 16.12	100	16.95	99
TKN	8.442	68	10.13	62	8.442	68	7.879	70	6.19	77	10.69	60
TP	2.2	96	4.3	92	3.7	93	4.7	91	0.3	99	0.7	99
TSS	7.4	99	4.2	99	3	100	5.6	99	6.4	99	6.2	99
Sulphate	51.67	28	41.56	42	43.78	39	56.12	22	47.15	34	51.1	29
Chloride	197.5	18	164.2	32	151.3	37	169.5	29	177.4	26	129.5	46
MLSS-2400 n	ng/L		7.02		9 17		8.24		9.41		0 56	
рп FC	7.00	_	0.68	_	0.17 2.23	_	0.34	_	0.41	_	8.50 1.56	_
BOD	42.74	98	28.17	99	29.24	98	32.3	98	34.9	98	49.37	97
COD	129.5	96	97.15	97	97.15	97	146.3	96	129.5	96	145.7	96
TKN	14.07	47	13.51	49	15.19	43	13.78	48	11.81	55	11.25	58
TP	9.8	81	7.7	85	8.2	84	7.1	86	6.3	88	6.7	87
TSS	14.2	98 22	11.1	99	10.2	99 52	12.2	99	10.4	99	9.4	99
Chloride	48.70	32 41	41.30	43 43	33.0 151.2	55 37	47.72	34 26	51.20 107.7	29 18	55.19 173.5	20 28
MLSS-2900 n	ng/L	71	155.0		131.2	51	170.5	20	1)/./	10	175.5	20
pH	8.22	_	8.71	_	8.61	_	8.69	_	8.68	_	8.48	_
EC	1.341	_	1.765	-	1.896	-	1.979	-	0.298	-	1.77	-
BOD	43.8	98	62.4	97	42.42	98	34.11	98	45.26	98	31.84	98
COD	146.3	96	195.1	95	146.3	96	113.7	97	146.3	96	113.7	97
TKN	10.13	62	7.87	70	7.31	72	6.19	77	9.57	64	10.69	60
TSS 1P	0.3 11.3	88	2.1	96	1.3	97	1.2	98	1 11	98	12	98 100
Sulphate	52.18	99 27	48 57	32	9.0 43.12	99 40	50.65	30	54 84	99 24	33.56	53
Chloride	186.5	22	197.7	18	196.5	18	201.3	16	204.8	15	167.5	30
MLSS-3200 n	ng/L											
pН	8.01	-	7.99	-	8.14	-	8.12	-	7.81	-	7.98	-
EC	0.356	-	1.713	-	0.566	-	1.343	-	0.566	-	1.462	-
BOD	54.56	97 97	49.56	97 97	43.78	98	45.56	98	34.54	98 97	31.56	98 97
COD	163.4	96 44	131./	96 52	114.0	97	121.0	97 55	106.7	97	104.5	97
TP	54	44 90	68	32 87	61	49	4.2	92	3.3	04 94	9.80	96
TSS	9.3	99	9.6	99	8.5	99	8.9	99	10	99	8.8	99
Sulphate	49.23	32	51.87	28	41.78	42	43.33	40	38.32	47	41.4	42
Chloride	178.3	26	175.8	27	167.9	30	189.3	21	165.8	31	176.8	26
MLSS-3700 n	ng/L		= 0.1		0.00		0.40		= 0 =			
pH	7.87	-	7.91	-	8.03	-	8.12	-	7.95	-	8.2	-
EC	0.545	- 07	1.434	- 07	1.705	- 08	0.038	- 08	1.430	- 08	0.407 367	- 08
COD	131.5	96	142.5	96	118.5	97	102.3	97	86.5	98	98.56	97
TKN	12.89	51	14.78	44	12.93	51	11.78	56	10.12	62	9.78	63
TP	7.6	85	7.8	85	6.4	88	6.9	87	5.3	90	7.6	85
TSS	9.6	99	8.8	99	8.5	99	8.1	99	7.5	99	8.7	99
Sulphate	54.56	24	49.44	31	43.55	39	49.7	31	34.5	52	41.52	42
MI SS-4200 m	134.6	44	1/0.0	26	105.5	31	186.4	22	189.4	21	154.3	30
pH	7.79	_	7.99	_	8.02	_	7.97	_	8.1	_	7.61	_
EC	0.302	_	1.714	_	0.335	_	0.321	_	1.565	_	1.464	-
BOD	46.63	98	27.2	99	21.85	99	20.54	99	19.44	99	16.19	99
COD	129.5	96	97.15	97	80.96	98	73.45	98	69.33	98	64.76	98
TKN	9.56	64	13.51	49	16.32	38	13.67	48	10.89	59	11.8	56
TP	7.8	85	6.8 5.9	87	4.4	92	5.4	90	6.2	88	4.8	91
155 Sulphate	8.0 41.26	99 43	53.56	99 26	7.8	99 28	0.5	99 37	5.5 40.22	99 44	7.4	99 55
Chloride	162.5	32	176.3	20	227.1	20	189.3	21	176.5	26	127.1	47

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GC-MS analysis: The GC-MS analysis was done for raw dairy effluent and for treated effluent using pretreatment techniques and SBR. Some of the compounds present in the raw effluent were *p*-cresol, dimethyl benzaldehyde, benzene, indole, *n*-decanoic acid, 3-methyl indole, dodecanoic acid, tetradecanoic acid, pentadecanoic acid, dibutyl phthalate,

n-hexadecanoic acid, oleic acid and octadecanoic acid (Fig. 2). After the treatment, several compounds were removed except dimethyl benzaldehyde, benzene, dibutyl phthalate and *n*-hexadecanoic acid (Table-3 and Fig. 3).

The compounds present in the effluent were determined by knowing the molecular weight. These compounds are





TABLE-3 GC-MS FOR DAIRY EFFLUENTS											
	Raw effluent		MLSS-2100 mg/L		MLSS-2400 mg/L		MLSS-2900 mg/L		MLSS-4200 mg/L		
Parameter	R.T.	Area	R.T.	Area	R.T.	Area	R.T.	Area	R.T.	Area	
	(min)	(%)	(min)	(%)	(min)	(%)	(min)	(%)	(min)	(%)	
p-Cresol	5.745	2.75	-	-	-	-	-	-	-	-	
Dimethyl-benzaldehyde	7.788	1.32	7.786	25.1	7.786	25.19	7.786	31.81	7.788	42.4	
Benzene	8.397	0.17	8.396	3.77	8.396	4.36	8.395	4.76	8.396	5.52	
Indole	9.112	0.12	_	-	-	-	_	-	_	-	
Dibutyl phthalate	17.512	6.89	_	-	17.513	4.6	17.512	3.4	17.512	3	
n-Hexadecanoic acid	17.664	52.49	17.428	5.62	-	-	_	-	-	-	





TABLE-4												
PERCENTAGE CHANGE IN AREA												
	Raw effluent		MLSS-2100 mg/L		MLSS-2	400 mg/L	MLSS-2	900 mg/L	MLSS-4200 mg/L			
Parameter	R.T. (min)	Change area (%)	R.T. (min)	Change area (%)	R.T. (min)	Change area (%)	R.T. (min)	Change area (%)	R.T. (min)	Change area (%)		
Dimethyl-benzaldehyde	7.788	-	7.786	94.75	7.786	94.75	7.786	95.85	7.788	96.88		
Benzene	8.397	-	8.396	95.49	8.396	96.1	8.395	96.42	8.396	96.92		
Dibutyl phthalate	17.512	-	-	-	17.513	33.23	17.512	50.65	17.512	56.45		
n-Hexadecanoic acid	17.664	_	17.428	89.29	-	-	-	-	_	-		

indicated at a particular retention time and percentage of area. The loss of compounds in treated effluent is indicated by the percentage change in the area at the respective retention time (Table-4).

Kinetic studies: Kinetic studies were performed for COD removal. The nature of biochemical reactions in wastewater is complicated and their composition will differ. Therefore, the following equation was used to determine k and K_s :

$$\frac{X\theta c}{S_o - S} = \frac{K_s}{k} \left(\frac{1}{s}\right) + \frac{1}{k}$$

where, k = maximum substrate utilization rate and used to find out the volume of the reactor. If the value of k is higher then the size of the reactor will decrease, K_s has no direct application in process design. It gives the idea about the change in specific growth rate of bacteria with change in the concentration of growth limiting substrate, Y is used to find the total amount of sludge produced in a wastewater treatment plant, K_d is used to find the net amount of sludge to be handled and hence the cost of sludge handling facility can be found out. And the X is the MLSS concentration in mg/L, S₀ and S are influent and effluent BOD, respectively. The above results indicated that at MLSS = 2100 mg/L is having better removal efficiencies than other. For MLSS = 2100 mg/L, the value of k and K_s was found to be 8 h⁻¹ and 4.85 mg/L, respectively. The k value affects the volume of reactor. Higher the value of k, smaller will be the size of the reactor. The k studied was found to be very high, indicating that a small size reactor is enough for the treatment of dairy effluent.

Conclusion

The pollution load and the presence of nutrients in the dairy industry are high when compared to the effluents from the other food industry. Therefore, these effluents must be treated before being released into waterbodies. In this work, the pretreatment of dairy effluent was performed by employing the advanced oxidation process (AOP) followed by a sequencing batch reactor as biological treatment. The initial characteristics of collected dairy effluent were performed according to APHA methods. The pretreatment techniques were already optimized for a combination of ozone and hydrogen peroxide. The ozone was passed for 5 min at pH 10 and then 10 mL H_2O_2 was added. The SBR was adapted with seed for developing the bacterial mass (MLSS) for the dairy effluent. The removal efficiencies of COD, BOD, TKN, TP, TSS, chloride and sulphate have to be evaluated for MLSS of 2100, 2400, 2900, 3200, 3700 and 4200 mg/L for the aeration time of 4, 6, 8, 12, 16 and 24 h, respectively. The percent removal of the above parameters

was found better for the MLSS of 2100 mg/L at 16 h aeration time were in the order of 100, 100, 77, 99, 99, 26 and 34, respectively. The performance of pretreatment was done for increasing the percentage removal efficiencies of pollutant load and nutrients.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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