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Physicochemical and Microbial Characterization of Treated and Untreated Produced Water

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Authors' contributions

This work was carried out in collaboration between both authors. Author LETA designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Author AJE managed the analyses of the study and the literature searches. Both authors read and approved the final manuscript.

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ABSTRACT

Introduction: The discharge of produced water into the aquatic environment presents a risk to the environment. This is a form of pollution and may release toxicants that are highly noxious to sensitive marine species even at low concentrations, which causes bio-degeneration/transformation and biodiversity loss.

Research Gap: There are insufficient literatures on extensive monitoring of the physicochemical and microbial characteristics of produced water. Existing literatures concerning analysis of treated and untreated produced water are not comprehensive with respect to number of physicochemical variables analyzed.

Aim: To ascertain if the physicochemical properties and microbial population of produced water were within acceptable regulatoy specifications.

Place and Duration of Study: The study was conducted in the Federal University of Petroleum Resources, Effurun, Delta State, Nigeria from July, 2021 – April, 2022.

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Methodology: Electrical conductivity, dissolved oxygen, pH, temperature, turbidity, total petroleum hydrocarbons, nitrates, phosphates, sulphates and others were assessed using standard methods. Microbial counts were carried out for total heterotrophic bacteria and hydrocarbon utilizing bacteria using the pour plate method.

Results: Values obtained for some key physicochemical parameters monitored in the produced water are as follow: pH (8.10±0.03 for untreated and 8.27±0.01 for treated); electrical conductivity $(35700±11 \text{ }\mu\text{s/cm}$ for untreated and $41600±17 \text{ }\mu\text{s/cm}$ for treated); total dissolved solids $(22848±14$ mg/l for untreated and $26629±9$ mg/l for treated); dissolved oxygen $(2.05±0.01$ mg/l for untreated and 4.23±0.03mg/l for treated); biochemical oxygen demand (28.90±0.7mg/l for untreated and 18.4 ± 0.1 mg/l for treated); phosphate $(2.05\pm0.01$ mg/l for untreated and 4.23 ± 0.03 mg/l for treated); nitrate (54.82±1.9mg/l for untreated and 50.21±0.9mg/l for treated); total hydrocarbon content (118.00±0.00mg/l for untreated and 34.00±0.00mg/l); total heterotrophic bacteria (4.1e+04 CFU/ml for untreated and 2.8e+04 CFU/ml for treated); and hydrocarbon utilizing bacteria (2.44e+03 CFU/ml for untreated and 1.58e+03 CFU/ml for treated). Statistical analysis of the produced water showed varying forms of correlation between physicochemical parameters of untreated and treated produced water.

Conclusion: It is important to assess properties of produced water before disposal into aquatic environment as chronic impact associated with long-term exposures may pose potential ecological risks.

hydrocarbon utilizing bacteria load.

Keywords: Produced water; hydrocarbons; physicochemical variables; heterotrophic and

1. INTRODUCTION

"Produced water is water that comes out of the well with crude oil during crude oil production. It contains soluble and non-soluble oil/organics, suspended solids, dissolved solids and various chemicals used in the production process" [1]. "In Nigeria, the petroleum industry depends majorly on the physicochemical analysis of produced water to monitor and regulate produced water discharge. This strategy has proved inappropriate and inadequate to protect aquatic organisms because it only gives information on the constituents and concentrations of the individual components in the produced water rather than their potential ecological risks/effect (biological interpretations) on aquatic organisms exposed to it" [2].

"The inorganic content of produced water is highly related to the geochemical characteristics of the well from which its produced. They present as dissolved salts, naturally occurring radioactive materials and heavy metal. Cations such as Na⁺, K⁺, Ca²⁺, Mg²⁺, Ba²⁺, Sr²⁺, Fe²⁺ and anions such as CI, SO_4^2 , CO_3^2 , HCO³⁻ affect produced water chemistry in terms of buffering capacity, salinity, and scale potential mainly due to dissolved sodium and chloride and also to a lower extent to calcium, magnesium and potassium, which may vary from a few parts per million to about 300000 mg/L" [2].

"Lesser volumes of heavy metals such as cadmium, chromium, copper, lead, mercury, nickel, silver and zinc mostly occur naturally. Lead is a toxic metal that enters the body through ingestion, inhalation, and skin absorption and can be accumulated in tissue. This affects most organs in human body especially kidneys and brains. Chromium is also toxic and water contaminated with chromium, results to skin irritation, livestock death, etc. Their concentration can reach 10^2 to 10^5 times the one found in seawater" [3].

"Naturally occurring radioactive materials (NORM) originating from the geological formation and are brought to the surface as dissolved solids in produced water. NORM may precipitate into scale or sludge when water temperature reduces as it reaches the surface. The most abundant NORM compound is 226 Ra and 228 Ra and barium and this is derived from the radioactive decay of uranium-238 and thorium-232 associated with certain rocks and clay in the hydrocarbon reservoir. When radium decays, it emits alpha and gamma rays, and exposure to radium causes cancer" [4].

"Production solids are a wide range of solid organic and inorganic materials that accompany produced water. They include formation solids, particulate detached from the surrounding materials, corrosion and anti-scale products from pipes and equipment, bacteria, waxes, and asphaltenes. Other inorganic crystalline substances such a $SiO₂$, $Fe₂O₃$, $Fe₃O₄$ and BaSO⁴ can also be found. The management of produced water is considered to be cost effective in the oil and gas field" [5].

According to Estrada and Viana [6] and [7], "there are different technological choices available, selection of a management option for produced water varies according to different factors such as chemical and physical properties of the water, flow rate of the water generated, end-us, regulations, technical and economic feasibility, etc". There are three major pollution prevention hierarchies that have been employed in the management of produced water according to the environmental preferences. They are:

- Minimization: used to minimize the production of produced water
- Recycle/Re-use: used to recycle produced water for other activities like irrigation for agricultural purposes.
- Disposal (when the produced water cannot be managed through minimization, re-use, or recycle).

Produced water comprises of different contaminants with varying concentrations, therefore, there are numerous treatment technologies available and proposed for the treatment of produced water. The best and effective way of handling produced water is to ensure that proper treatment is done [8]. Produced water treatment has the potential to produce harmless and valuable product rather than a waste, and it could be used for irrigation, various industrial uses and livestock/wildlife watering or power plant make-up [9]. A wide variety of produced water treatment methods have been reported previously [10]. The treatment system always requires a series of individual unit processes for contaminant removal that might not be removed through a single process.

Treatment of produced water can help in facilitating additional options for water management including its re-use for agricultural and industrial purposes. As cited by Arthur et al. [11], the final disposal of water is determined by the type and extent of treatment of produced water from the onshore oil and gas production operation treatment facilities which are designed to remove dispersed oil and grease and suspended solids, to avoid plugging and pumps

damage. While in offshore operations, the main treatment objective is to reduce the oil and grease to an acceptable level and mitigate toxic impacts on aquatic fauna and flora. It is a common practice to discharge the treated produced water into the sea [12].

In general, produced water treatment process has three main stages, pre-treatment, main treatment step, and final polishing treatment step. The pre-treatment step is done to remove large volume of oil droplets, coarse particles and gas bubbles to reduce dispersed contaminants. The main treatment step involves primary treatment in which small oil droplets and particles removal will be achieved and will be done by using skim tanks, plate packs interceptors and API separators. The secondary treatment will involve removal of much smaller oil droplets and particles using gas flotation, hydro-cyclones and centrifuges.

According to Viana et al. [7], the combination of physical, chemical and biological treatment processes should be used for the achievement of the different treatment goals.

The major objectives for operators of produced water treatment are as follows:

- Removal of free and dispersed oil and grease present in produced water
- Removal of dissolved organics
- Removal of microorganisms, algae and bacteria
- Removal of turbidity via elimination of suspended particles and colloids
- Removal of dissolved gases
- Removal of dissolved salts and minerals, excess water-hardness and possible radioactive materials.

"Following the treatment of produced water, there have been an increasing attention on reclaiming, reusing and recycling of water that is usually wasted to meet the communities' needs of freshwater sources" [13]. Different standards have been developed for the reuse of treated water provided based on intended purposes. The United State Environmental Protection Agency (USEPA) standards provide limitation for reuse of treated water as drinking water [14], while the US Department of Agriculture Natural Resources Conservation Service provides the standards for reuse in irrigation and livestock [15] as shown in Table 1.

Component	Drinking (g/m3)	Irrigation (g/m3)	Livestock (g/m3)
Lithium		2500	
Potassium			
Sodium	200	Based on SAR	2000
Ammonium	1.5		$\overline{}$
Calcium	-	Based on SAR	$\overline{}$
Magnesium		Based on SAR	2000
Barium			٠
Chloride	250		1500
Bicarbonate			$\overline{}$
Sulphate	250		1500
Conductivity (dS/m)	$\overline{}$	2.5	$1.5 - 5$
Sodium Adsorption ratio (SAR)	$\overline{}$	$0 - 6$	۰.

Table 1. Standards for water reuse for drinking, irrigation and livestock purposes

Source: [15].Sodium adsorption ratio (SAR)

"Furthermore, there are several alternatives for the utilization of treated produced water such as drinking water, irrigation, livestock watering, habitat and wildlife watering, fire control, and industrial uses such as dust control, oil field uses, and power generation" [16].

The discharge of produced water into aquatic environment presents a risk to the environment. This is a form of pollution and may release toxicants that are highly noxious to sensitive marine species even at low concentrations, which causes bio-degeneration/transformation as well as biodiversity loss. Hence, this study was conducted to determine and reveal the overall health and status of the recipient environment by determining the physicochemical and bacterial parameters of untreated and treated produced water.

2. METHODOLOGY

2.1 Sources of Test Samples: Produce Water Effluent (Wastewater)

Samples of produced water (untreated and treated) were collected from an offshore operational facility situated in Akwa Ibom State with GPS coordinate 03'51.141N; 006'58.794'E. A 10 -liter plastic sampling container was used for sample collection for physicochemical parameters analysis and glass containers for samples for analysis of TPH. Sample for BOD was collected in amber glass bottles. Sterile plastic bottles were used to sample for microbial analysis of the test sample. Samples for physicochemicals and microbial counts were stored at 4° C prior to testing. Samples for TPH were preserved with 1:1 v/v of tetraoxosulpahte (VI) acid (H_2SO_4) , while samples for metals were

preserved with 1:1 v/v of trioxonitrate v acid $(HNO₃)$.

2.2 Physicochemical Analysis of Test Samples

Produce water samples were analyzed following the standard method of American Public Health Association [17].

2.2.1 *In situ* **parameters**

These *In situ* parameters were assessed on site using the following procedures; pH (APHA 4500- H + B using Hanna pH electronic meter), temperature (APHA 2550 - B laboratory and field methods), electrical conductivity and total dissolved solids (APHA 2510-B using Hanna desktop conductivity meter) and dissolved oxygen (DO) (APHA 4500-O C by azide modification method)

2.2.2 Laboratory analysis

These physicochemical parameters were assessed using the following procedures; Salinity (Mohr Argentometric Method, 4500 B-Cl-), Biochemical oxygen demand (BOD) (APHA 5210 B, by 5-Day test method), Total suspended solids $(APHA 2540D)$, Nitrates $(APHA 4500-NO₃ B)$ and Phosphate (APHA 4500-PE). Oil and grease and THCof the samples was analysed using ASTM D3921 method.

Isolation of heterotrophic bacteria in the produced water: The method of Chikere and Ekwuabu [15] was adopted. Nutrient agar (NA) was used for bacteria enumeration. The media were prepared according to manufacturer's specification. Ten-fold serial dilution was carried out using 1ml of the test sample and 0.85% (w/v) sodium chloride as diluent. The standard pour plate method was used by inoculating 0.1ml aliquot of the different dilutions into sterile Petri dishes and 15 ml- 20 ml of cooled media was poured into each of the plates. The culture plates were swirled for homogenization, allowed to solidify and incubated at $28 \pm 2^{\circ}$ C for 18-24 hours (bacteria) After incubation, individual colonies were recorded as colony forming unit (CFU/ml)

Isolation and selection of hydrocarbon bacteria: The procedure of Bhattacharya et al. [19] was adopted for this study. Bushnell-Haas (BH) media with the following composition (g/L) : K₂HPO₄ (1.0 g), $KH_{2}PO_{4}$ (1.0 g), $NH_{4}NO_{3}$ (1.0 g), $MgSO_{4} \cdot 7H_{2}O$ (0.2 g), FeCl₃•6H₂O (0.05 g), CaCl₂•2H₂O (0.02 g), was used as an enrichment medium with 1% crude oil (v/v) as the sole carbon source to isolate the crude oil degrading bacteria from the produced water samples. Soil samples (10g) were added to 50mL BH media in 250mL Erlenmeyer culture flasks. It was then incubated at 28 \pm 2°C for 7 days. After 7 days incubation, the bacteria cultures were isolated as single colonies into petri dishes containing nutrient agar (NA) media by streak-plate method. The pure bacteria isolates were maintained in slant cultures by preserving at 4°C until required for use.

2.3 Statistical Analysis

Statistical package for social sciences (SPSS) was used to analyze the different variables.

3. RESULTS AND DISCUSSION

3.1 Physicochemical Parameters of Test Samples

Result of physicochemical properties of treated and untreated produced water (wastewater) is presented in Table 2.

Physicochemical properties of wastewater in relation to permissible limits are seen in Figs. 1- 3. The overall effect of treatment is implicated in the difference in determining physicochemical parameters of treated and untreated wastewater. Fig. 1 shows the values for density, pH, dissolved oxygen and phosphate in treated and untreated wastewater against the regulatory limit [19].

The density of untreated wastewater (1.018894) changed slightly to 1.017778 at room temperature, with values slightly above potable water (0.99704). Nevertheless, density values have little or no effect on the quality of treatment. The pH values of untreated (8.10 ± 0.03) and treated (8.27 ± 0.01) are within permissible limits of 6.5-8.5 for wastewater as reported by WHO [18].

The pH values of treated and untreated wastewater are moderately alkaline. Dissolved oxygen (DO), an important parameter in accessing water quality, influences aquatic lives. DO in wastewater increased from 2.05±0.01mg/l to 4.23±0.03mg/l after treatment. Effect of treatment observably increased DO levels in produced water, exceeding permissible limits of 4mg/l. High DO values may indicate aeration (as introduced by the mechanical treatment process), biochemical (an autotrophic process of phytoplankton) or biological (decreased eutrophication due to treatment) processes taking place in wastewater. A decrease in eutrophication is most likely, as there is a considerable decrease in concentration (colony forming units, CFU) of total heterotrophic bacteria (THB) from 4.1×10^4 CFU/ml to 2.8 \times 10⁴ CFU/ml after treatment. Hydrocarbon Utilizing Bacteria (HUB) also reduced from 2.44 \times 10³ CFU/ml to 1.58 \times 10³ CFU/ml after treatment. Both THB and HUB were above the tolerable limits of 100 and 0 CFU/ml, respectively (see Table 1 and Fig. 2). Different researchers have reported the presence of high numbers of THBs and HUBs in environments containing hydrocarbons as cited in [21]. Temperature and pressure have been proven to correlate with DO [22]. Research has shown that temperature is inversely proportional to DO and increases with pressure.

Electrical conductivity, a determinant of the total ionized constituent of water, is directly proportional to the sum of the cations and anions [23]. Phosphate content (mg/L) was found to be higher (7.56±0.06) in untreated wastewater than treated (3.29±0.09), and the phosphate content in treated wastewater was within tolerable limits (3.5 mg/L). The removal of anthropogenic phosphorus from wastewater during treatment has a considerable effect on the phosphate content of wastewater. High values of phosphate in wastewater represent high pollution loads and cause eutrophication of the aquatic body [24]. The reduced value of phosphate in treated water can be corroborated by high oxygen availability (higher value of DO) as discussed above.

Electrical conductivity (EC), total dissolved solids (TDS), salinity, total heterotrophic bacteria (THB) and hydrocarbon utilizing bacteria (HUB) in untreated and treated wastewater in comparison with standard limits are graphically presented in Fig. 2. Interestingly, values for these five (5) parameters were above their respective permissible limits despite treatment. The EC contents changed from a mean value of 35700±11 µS/cm (untreated) to 41600±17 µS/cm (treated). Though EC values are higher than the maximum permissible limit of 1000 uS/cm, these high values were similar to the content reviewed by Johnson et al. and Annapoorna et al. [24,25]. As identified by Amiri et al. [26], "the EC values show a high correlation level with many of the water quality parameters, especially total dissolved solids, (TDS), salinity (chlorides), total alkalinity, sulfates concentration, total hardness and magnesium concentration". "Water with high EC values may not necessarily pose a risk to human health, but it can cause corrosion in industrial equipment or plumbing systems, scale build-up, mineral-like taste in drinking water, and pose challenges with a dissolved solid concentration in agriculture" [27].

The observed total dissolved solids (TDS) in the untreated and treated wastewater changed from 22848±14 mg/l (untreated) to 26629±9 mg/l (treated). These values are higher than the

permissible limits of 2000 mg/l for wastewater. Observably, TDS increased when treated, possibly due to the treatment method adopted by the industry. The high value of TDS influences the tastes, hardness and corrosive properties of water [28]. The salinity in the untreated water (9529±13mg/l) increased to 11104.87±7.58mg/l when treated. These values were found above permissible limits of 600mg/l for wastewater.

The oil and grease, total hydrocarbon content (THC), temperature, biological oxygen demand (BOD), turbidity and nitrate values for treated and untreated wastewater are presented in Fig. 3. Apart from turbidity, the effect of treatment is apparent in all six (6) physicochemical parameters as their values decreased considerably after treatment.

The study showed that oil and grease (OG) concentration reduced from 125±1.5mg/L to 47±1.1mg/L after treatment, with both values exceeding the NUPRC permissible limits of 10mg/L. The presence of OG is important to water quality and safety assessment. High OG levels in water can trigger surface films and shoreline deposits, which leads to environmental degradation, and can induce human health risks when discharged into surface and groundwater. "In addition, OG may interfere with anaerobic and aerobic biological and biochemical processes, leading to decreased wastewater treatment efficiency" [29].

Fig. 1. Physicochemical parameters (density, pH, DO, phosphates) of treated and untreated wastewater

Fig. 2. Selected physicochemical parameters and Microbial counts of treated and untreated produced water

The total hydrocarbon content (THC) of wastewater reduced considerably from 118±0.00 mg/L to 34±0.00mg/L after treatment. However, THC values of untreated and treated wastewater were found to be considerably higher than permissible limits of 0.7mg/L, indicating an inadequate treatment process. Hydrocarbon contents (HC) in drinking water lead to unacceptable taste and odor. "Further, the effect of HC in water could have narcotic properties and may lead to irreversible effects on the nervous system. Aromatic hydrocarbons are

generally, more polar than aliphatic hydrocarbons and therefore tend to be more soluble in water and less volatile than aliphatic hydrocarbons with a corresponding number of carbon atoms" [30].

The temperature of wastewater in the study was found to reduce from 28.3°C (untreated) to 27.6°C (treated), with both values within the permissible limit of <40°C. A decrease in the temperature of water decelerates chemical reactions, increases the solubility of gases, alters

taste and odour and depresses the metabolic activity of organisms [31].

There is a significant decrease in the biological oxygen demand (BOD) of wastewater from 28.9±0.7mg/L (untreated) to 18.4±0.1mg/L (treated). The BOD values of both untreated and treated wastewater were found to be above the permissible limits of 10mg/L. Low BOD was mainly due to higher productivity of phytoplankton, along with increased oxygen solubility at reduced temperatures, while high BOD values resulted from the rapid utilization of oxygen at higher temperatures [32].

The turbidity (Nephelometric turbidity unit, NTU) of the wastewater samples increased from 31.24±0.27 (untreated) to 34.33±0.35 (treated), which are all above the permissible limits of 15NTU for wastewater. High turbidity levels may affect disinfection systems and prevent pathogen removal during treatment. It may also indicate microbial presence [27]. Reduced Turbidity is one of the major indicators for effective water treatment process. The increased turbidity after treatment indicates that the treatment process is insufficient as it showed negative effect on the turbidity of treated wastewater. Further, the ineffectiveness of the treatment process can be observed in other physicochemical parameters which showed slight decrease, but should have originally been reduced to a tolerable limit.

Nitrate concentration (mg/L) from the study reduced from 54.82±1.9 (untreated) to 50.21±0.9, with both values slightly exceeding the permissible limit of 50mg/L for drinking water. Nitrate, an important source of nitrogen for plant and animal life, can be harmful to life if permissible limits are exceeded. Gases from refining activities contain CO, NO and hydrocarbons which react in the sunlit atmosphere to produce $NO₂$, $O₃$, peroxyacetyl nitrate (PAN), atmospheric aerosols and other compounds. Nitrates find their way to wastewater affecting human health [33].

3.2 Correlation Analysis of Parameters

To assess the relationship amongst physicochemical parameters of the wastewater samples, correlation coefficients were worked out and a number of significant correlations were obtained. Table 3 showed the correlation matrix of the 17 physicochemical variables. It is clear from the results that DO as well as temperature were negatively correlated with all the variables and were not significantly correlated with any of the studied parameters except to themselves, with a very weak positive correlation of 0.139. All the variables except DO were positively and significantly correlated (at 0.05 level) with all the studied parameters.

Fig. 3. Physicochemical parameters (OG, THC, temperature, BOD, turbidity, nitrate) of treated and untreated produce water

Table 3. Correlation matrix of physicochemical parameters of treated and untreated produce water

The test of significant difference between treated and untreated wastewater was found to be significant at 5% level with values in Table 4. There was no significant difference found between the variables of treated and untreated wastewater. According to Amiri et al. [26] the EC finds higher level correlation significance with many of the water quality parameters, like TDS, chlorides, total alkalinity, sulphates, total hardness and magnesium. Similarly, Chikwe et al. [34] identified that all the parameters are more or less correlated with others in the correlation and regression study of the physiochemical parameters of ground water. The water quality of ground water can be predicted with sufficient accuracy just by the measurement of EC alone [14]. Nevertheless, records show low level of prediction in wastewater, as physicochemical properties are usually dependent on nature and type of wastewater treatments as they are to nature of effluents. The predictability of physicochemical parameters of wastewater provides a means for easier and faster monitoring of water quality in a location. Nwabueze et al. [35] concluded that the correlation study and correlation coefficient values can help in selecting treatments to minimize contaminants in groundwater.

4. CONCLUSION

Biological monitoring of produce water physicochemical parameters of treated and untreated produced water effluent were conducted to rule out the possibility of the presence of harmful chemicals which possess threats to aquatic and terrestrial life. Values obtained for some key physicochemical parameters monitored in the produced water effluent are as follows: density (1.018894± 0.0000mg/L for untreated and 1.017778± 0.0000mg/L for treated); pH (8.10±0.03 for untreated and 8.27±0.01 for treated); electrical conductivity (35700±11 µs/cm for untreated and 41600±17 µs/cm for treated); total dissolved solids (22848±14mg/L for untreated and 26629±9mg/L for treated); dissolved oxygen (2.05±0.01mg/L for untreated and 4.23±0.03mg/L for treated); biochemical oxygen demand (28.90±0.7mg/L for untreated and 18.4±0.1mg/L

for treated); phosphate (2.05±0.01mg/L for untreated and 4.23±0.03mg/L for treated); salinity (9529±13mg/L for untreated and 11104.87±7.58mg/L); nitrate (54.82±1.9mg/L for untreated and 50.21±0.9mg/L for treated); total hydrocarbon content (118.00±0.00mg/L for untreated and 34.00±0.00mg/L); total heterotrophic bacteria (4.1e+04CFU/mL for untreated and 2.8e+04CFU/mL for treated); and hydrocarbon utilizing bacteria (2.44e+03 CFU/mL for untreated and 1.58e+03 CFU/mL for treated). In addition, statistical analysis of physicochemical parameters and heavy metal concentration of wastewater showed varying forms of correlation between physicochemical parameters of untreated and treated wastewater.

Remarkably, values for five (5) of these parameters fall above their respective permissible limits despite treatment. The oil industry from which this research is conducted as well as other similar industries should improve on their treatment processes as findings showed treatment procedures are apparently insufficient. Without proper treatment and disposal of produce water into shallow estuarine and marine waters, some metals and higher molecular weight aromatic as well as saturated hydrocarbons may accumulate in sediments, bioaccumulate in bottom living biological communities which shall possess threat to the health of animals and by extension humans who are the final consumers.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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