



# Sustainable Production of Slow Release Fertilizers for Eco-Nutrient Delivery to Soil Microbial Communities in Nutrient Deficient soils

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## Authors' contributions

*This work was carried out in collaboration among all authors. Author CBEE conducted the study, performed the analysis, wrote the protocol, and wrote the first draft of the manuscript. Author CBC and Author AO conceptualized and supervised the research. All authors read and approved the final manuscript.*

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## ABSTRACT

There is a growing need to develop a natural-based slow-release fertilizer for sustained nutrient delivery as a result of the challenges associated with the direct application of chemical fertilizers. In this study, two low crop residues, cassava mesocarp CM and coconut coir dust CD were employed in the formulation and production of slow-release fertilizers (SLR). The proximate and mineral content analysis revealed an appreciable quantity of micronutrients and other soil stabilizers in the crop residues. Super absorbent polymers produced from both crop residues (CM-SAP and CCD-SAP) revealed water absorption positive correlation values as 0.9842 and 0.9859 and final dry weight values ranging from 2.66–2.99g and 2.20–2.28g, respectively. NPK garden fertilizer (20:10:10) was encapsulated in to each SAP to create the slow- release properties of the products branded casmes and coco dust. The conductivity analyses performed on the SLR formulations showed a steady release of the nutrient content in to the liquid medium which ranged from 10µs/cm at 30mins to 290µs/cm at 160mins for casmes while cocodust had a final value of 110µs/cm. Both

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formulations were applied as biostimulants alongside other nutrient regimens and it was observed that the products supported the growth of active bacteria population competing favourably with other biostimulants.

*Keywords: Slow release fertilizer; bioremediation; biostimulants; polymer.*

## 1. INTRODUCTION

For so many years, the aim of applying fertilizers in the environment has been to provide nutrients for plants and microorganisms to increase and sustain optimal crop yield and for microbes to perform their requisite metabolic functions. The source of nutrient (usually nitrogen and phosphorus) could be organic or inorganic. However, any fertilizer irrespective of the source can be harmful to the environment if misused.

In recent times, fertilizer has been labelled by environmentalists as a source of pollution for soil, water and air environments. Although inorganic nutrient sources such as NPK,  $K_2HPO_4$ ,  $NO_3-N$ , have been reported to be successful in cleaning-up crude oil both on land and water by stimulating the growth of remediating microbes [1] and increasing crop yield. However, the direct application has been linked to nitrate leaching in groundwater, emission of greenhouse gases (nitrous oxide), soils polluted with toxic heavy metals and surface run off N and P nutrients causing aquatic eutrophication [2]. Similar findings have been reported across the world that a great percentage of nitrogen derived from fertilizers are not taken up by plants or microbes which may become immobilized in soil organic matter or may be lost to the environment. Loss of N to the environment usually occurs when high concentrations of soluble N forms are present in soil solution in excess compared to the amount that plants can take up at a particular time [3].

To ensure that proper use of fertilizer is beneficial to both crop production, microbial metabolism and generally the environment, researchers and fertilizer producers have designed means of achieving defined goal of fertilizer use, that is by improving fertilizer use efficiency and minimizing negative environmental impacts, however much have not been achieved. However, these problems can be effectively overcome with good management practices which include selecting a rate application system compatible with plants and microbial needs per time. The use of slow-or controlled-release fertilizers presents an effective management tool owing to its tremendous advantages in terms of

labour saving, increased nutrient uptake, reduced fertilizer application rates, potential for improved yields and reduced environmental impacts. Controlled release fertilizer (CRF) is a purposely designed nutrient delivery system that releases active fertilizing nutrients in a controlled, delayed manner in synchrony with the sequential nutritional needs of organisms and plants, thus, they provide enhanced nutrient use efficiency along with enhanced growth capacity [4, 5, 6]. Superabsorbent polymers (SAP) are required in the formulation of controlled/slow release fertilizers which are responsible for the water absorption and retention properties. Interestingly, there are certain natural based polymers with excellent super absorbent properties that have caught the interest of research circles, some of which was considered in this study. The potentials of natural based polymers with all its environmental advantages have not been explored especially in Nigeria.

Currently, most of the SAP used in practice are mainly petroleum-based synthetic polymers such as polysulfone (PSF), cellulose acetate (CA) and polyacrylonitrile (PAN), which have high production cost and poor environmentally friendly characteristics hence the development of natural polymer based super absorbents has become subject of great interest having commercial and environmental advantages [7, 8]. Presently, many natural polysaccharides such as guar gum, cellulose, chitosan, starch, alginate and their derivatives modified with synthetic polymers have been adopted to prepare new types of super absorbents [8]. However, starch has recently caught the attention of researchers because it occurs naturally as a polysaccharide biopolymer that is abundantly available from many renewable plant sources. Due to its slow cost, biodegradability and abundance, several non-food applications of starch have been investigated [9] with starch based controlled release coating material as one of the numerous applications [10, 11]. Other biopolymers such as cellulose, alginate, wheat gluten and rubber have been explored for their suitability in enhancing the efficiency of fertilizer release [12]. The advantages of these biopolymers over petroleum-based polymers are their

sustainability, biodegradation properties and base components that are non-toxic [13].

Therefore, this research was designed to develop a sustained nutrient delivery system using natural based polymer coating materials with properties suitable for withstanding environmental variations, supporting environmental sustainability and efficiently delivering nutrients over a long period of time synchronized with the requirements of plants and microbes in the soil

## 2. MATERIALS AND METHODS

### 2.1 Sourcing and Preparation of Samples

Coconut coir dust, cassava mesocarp powder and cassava starch were obtained from coconut husk, cassava processing meal and local market, respectively. Cassava mesocarp peel was washed, sun dried and ground in a cassava grinding machine to obtain a fine powder. To produce coconut coir dust, dried coconut husk was shredded and a large quantity of the dust was generated which was gathered together and sieved using a 50–100 $\mu$ m sieve to obtain particles in this range. This was stored in an air tight plastic container prior to use as a super absorbent polymer.

### 2.2 Proximate Analysis

Proximate analysis is a quantitative analysis used to determine the different micronutrient components in an organic material. In this study, ash (furnance method), moisture (air oven method) lipids (soxhlet extraction method), carbohydrate (Cleg Anthrone method), fibre (standing method) and protein (Kjeldah method) were determined in each organic compound according to [9] and [11]. Mineral content, phosphate and nitrate were also determined by Ascorbic and Brucin methods, respectively.

### 2.3 Super Absorbent Formulation

Samples of cassava mesocarp powder (CSM), coconut coir dust (COD) and cassava starch were used in formulating the super absorbent polymer. For the preparation, 12.4g and 134g of CSM and COD respectively were weighed into two separate bowls. Gelatinized cassava starch was prepared by making a paste with cassava powder and water after which boiled water was added until it thickened. Prepared cassava starch

was added to CSM and COD and mixed thoroughly until both mixtures blended. Then, 2ml of vinegar was added to both mixtures as a preservative. Each formulation was rolled into balls which ranged from 4.80–4.90g in mass and dried. Dry weight analysis was conducted to determine the weight loss pattern of all formulations.

### 2.4 Measurement of Water Absorbency of CRF

Accurately weighed CRF (about 0.5g) was immersed in 10ml of tap water and allowed to soak at room temperature for 2h. The swollen capsule was filtered through an 80-mesh sieve to remove non absorbed water and weighed. The water absorbency was calculated using the following equation:  $WA = (M - M_0) / M_0$

Here M and M<sub>0</sub> denote the weight of swollen CRF and the weight of the dry CRF, respectively, and WA is the water absorbency per gram of dried CRF.

### 2.5 Dry Weight loss Determination of Super Absorbent Polymers

Formulation of super absorbent polymer (SAP) was designed with two crop residues (cassava mesocarp and coconut coir dust) known for their excellent water absorption properties. After preparation, the SAPs were rolled into balls ranging from 4.87-4.90g and dried for a period of 10days to determine dry weight loss pattern. Readings were taken at two days interval to determine the weight loss pattern of each ball until a constant weight was achieved.

### 2.6 Slow release fertilizer formulation

Slow release fertilizer was prepared using the super absorbent polymers (SAP) as an encapsulating matrix for coating the in organic NPK20:10:10 fertilizer. While preparing the SAP, holes were burrowed into the SAP granules before drying and after which 3 granules of NPK fertilizer was inserted in to the SAP, sealed and dried again in a hot air oven. This was done for both SAP formulations. Fig. 1 presents the formulation model for both slow release fertilizers.

### 2.6 Conductivity Analysis

Conductivity analysis was determined on both SRF formulations (casmes and coco dust) to

determine the nutrient release rate over time in a liquid medium. Samples were immersed in deionised water for 160min and readings were taken at intervals of 10min using a conductivity meter.

## 2.7 Sample Collection/Preparation

Crude oil polluted soil was collected from an aged-spill site at Imo River Egbu, Kom kom community, Rivers State, Nigeria using a soil auger machine at different depths from top soil (0–20cm) at different points. Soil samples were homogenized by proper mixing and transported to the laboratory for analysis within 6h of collection.

## 2.8 Enumeration of total Culturable Heterotrophic and Hydrocarbon Utilizing Bacteria (THB and THUB)

From each treatment, 1g of soil (wet weight) was homogenized in 0.85% of normal saline using a Heindolph vortexing machine. Decimal dilutions (10-fold) of the suspensions were plated out on Plate Count Agar (Merck, Germany) and incubated at 30°C for 24h for the THB counts. For THUB counts, culture enrichment was done using 1g of soil inoculated into 100ml of Bushnell Haas Mineral Salts (BHMS) medium (Sigma-Aldrich, USA) supplemented with 0.5% crude oil (v/v) as the sole carbon source. The mixture was incubated at 37°C at 130rpm for 5days. Bacteria

from this culture were enumerated by total plate count method using serial dilution technique on BHMS agar medium supplemented with crude oil. The plates were incubated aerobically at 37°C for 5days [14]. Distinct colonies were enumerated and streaked on nutrient agar plates for sub-culturing for further analysis.

## 3. RESULTS AND DISCUSSION

### 3.1 Proximate and Mineral Content Analysis

Cassava mesocarp and coconut coir dust were prepared from cassava mesocarp (Fig. 2a) and coconut husk (Fig. 2b) and the proximate and mineral content analysis results are presented in Figs. 3a and b, respectively. The proximate analysis of the different components of the organic waste materials has helped in assessing its potential for utilization as a nutrient supplement and super absorbent polymer formulation. The high fiber content presenting both samples supports the basis for their water absorption potential.

Nitrate had a significantly higher concentration than phosphate in both samples although both were present at appreciable quantities. The benefits and potential applications of organic wastes can only be explored when the components and constituents are known. In

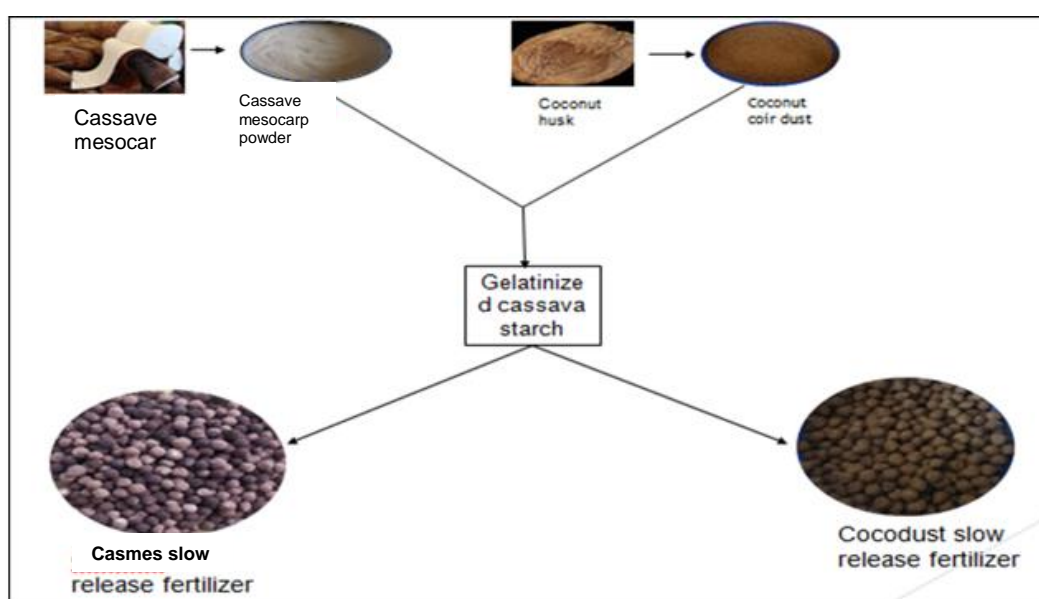


Fig. 1. Slow release fertilizer (casmes and cocodust) formulation model

general, the results obtained indicated that the crop residues are rich in rate-limiting nutrients (phosphate and nitrate) required to stimulate microbial growth and consequently enrich soil fertility. According to Agarry and Ogunleye [15] organic wastes act as both bulking agents and bacterial biomass suppliers.

### 3.2 Weight Loss Ratio of the Super Absorbent Polymers

The dry weight values experienced a gradual and steady decrease from day 2 to the end of the experiment at day 10. The weight loss rate for coconut coir dust derived SAP (COD-SAP) showed related trend however, the weight loss

rate was higher than CSM-SAP. Non-linear power model fitted best to the data generated for both CSM-SAP and COD-SAP. This model was used to determine the correlation coefficient  $R = -0.9864; -0.9762$  and coefficient of determination  $R^2 = 0.9731; 0.953$  for CSM-SAP and COD-SAP, respectively. The negative value observed in the correlation coefficient values resulted from the reduction in the water content during the drying process with time. The  $R^2$  values implies that the model can be used to make future predictions with 97% and 95% accuracy for CSM-SAP and COD-SAP, respectively. Results for dry weight loss analysis for both SAPs are illustrated in Fig. 4a and b



Fig. 2. Agricultural waste materials for super absorbent polymer production. A: Cassava mesocarp powder obtained from cassava mesocarp B: Coconut coir dust obtained from coconut husk

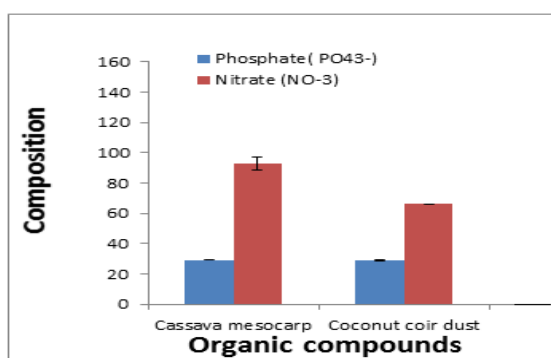


Fig. 3a. Mineral content composition of organic compounds in raw materials.

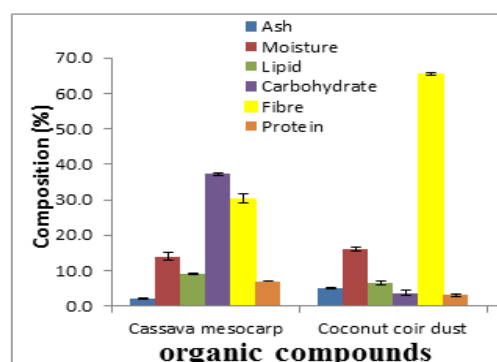


Fig. 3b. Proximate analysis of organic compounds in raw materials

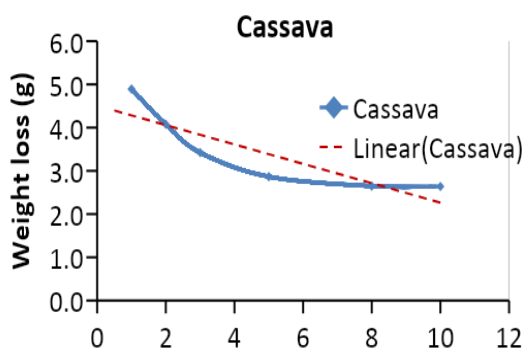


Fig. 4a. Dry weight loss (g) analysis of cassava mesocarp (CSM-SAP)

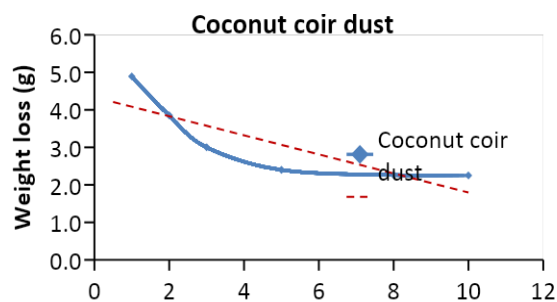


Fig. 4b. Dry weight loss (g) analysis of coconut coir super absorbent polymer (COD-SAP)

### 3.3 Water Absorption Potentials of the Super Absorbent Polymers

Water absorption analysis was conducted to determine the water absorption potential of each formulation at a particular time. The process was repeated and results were documented at intervals of 5 minutes for a period of 1 hour. The values obtained fitted best to the non-linear polynomial model.  $R^2$  values were 0.9686; 0.9680 while correlation coefficient  $R$  values were 0.9842; 0.9839 for CSM-SAP and COD-SAP, respectively. The positive correlation coefficient values obtained were as a result of increase in water concentration within the SAP granules with time. Fig. 5a illustrates the water absorption potential of both super absorbent formulations in parallel and CSM-SAP showed a significant higher water absorption capacity than COD-SAP. This could be attributed to the higher carbohydrate content presenting cassava. Chinma et al [16] proposed that the chemical compositions that enhance water absorption capacity are carbohydrates and protein owing to their hydrophilic constituents such as polar or charged side chains. Based on the success of water absorption analysis with cassava starch has binding agent, known concentrations of NPK fertilizer were encapsulated by the SAP using physical method to produce casmes and coco dust slow release fertilizers.

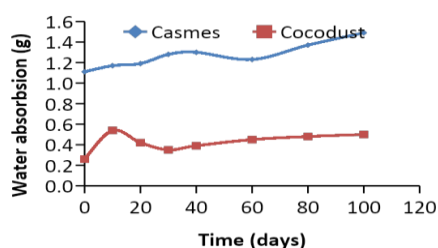
### 3.4 Sustained Nutrient Release Potential of Slow Release Fertilizers

Conductivity analysis was conducted to ascertain the rate of nutrient release per time before complete disintegration. Casmes, initially experienced a lag phase from 0–30 mins (conductivity reading was  $10\mu\text{s}/\text{cm}$ ) after which conductivity value increased progressively till

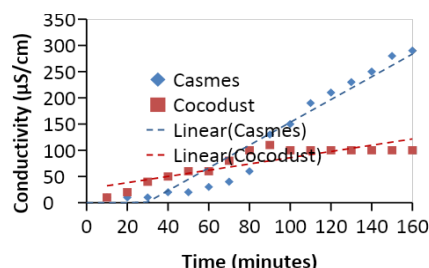
160min from  $10\text{--}290\mu\text{s}/\text{cm}$  while coco dust witnessed a slight variation at the beginning of the experiment compared to casmes Fig. 5b Linear quadratic model revealed  $R^2$  for both casmes and coco dust as 0.9650 (96%) and 0.9505 (95%), respectively. Conductivity analysis is a quick and simple method for determining the salt concentration of a medium and has been adopted by researchers as a nutrient monitoring tool in the environment [17]. Both slow release formulations increased the conductivity of the medium progressively but casmes had a higher conductivity value than coco dust. This observation can be likely interpreted that the binding agent (starch) had a stronger effect on casmes than coco dust with high fibre content which encouraged the higher rate of nutrient release. Starch has been confirmed a good binding agent in conventional tablets and capsules production due to its non-toxic and non-irritant properties as well as a diluent, disintegrant, and lubricant properties[18].

### 3.5 Heterotrophic and Hydrocarbon Utilizing Growth Profile

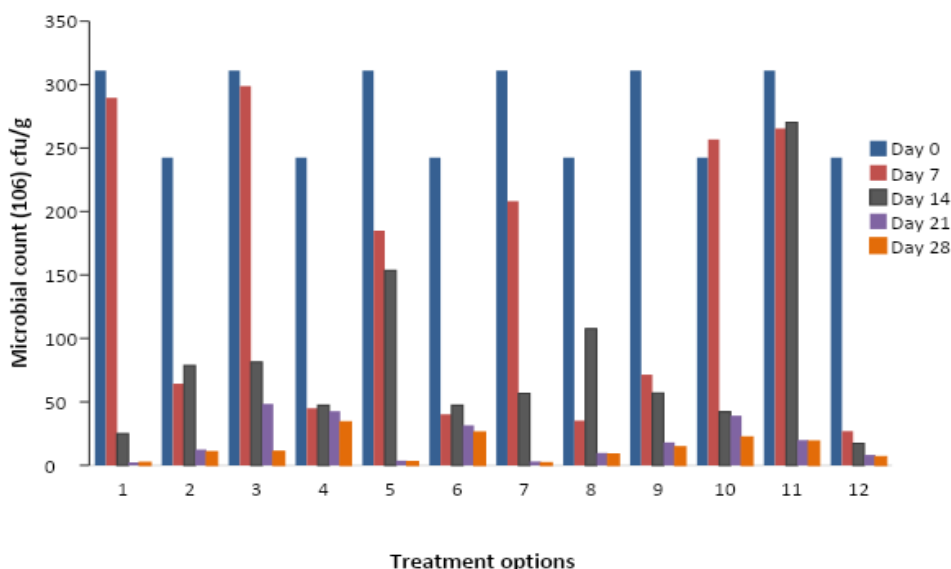
The bacterial population dynamics during the experimental period is presented in Fig. 6. As observed all treatment regimens had varying population dynamics at different days. However, the formulated nutrient amendments, CM and CCD competed favourably with other treatments and the results indicates that both SRFs supported a sustained nutrient delivery sustaining the growth of both THB and THUB populations in the polluted sample. For THB counts at day 7 all treatments had same order of magnitude ( $108\text{cfu}/\text{g}$ ) except NPK which was a magnitude less than others. CCD had highest heterotrophic bacterial population till day 21 and dropped slightly at day 35 while CM and other



**Fig. 5a.**Water absorption analysis of CSM-SAP and COD-SAP using starch as binding agent



**Fig. 5b.** Non-linear quadratic model for casmes and coco dust conductivity analysis



**Fig. 6.** Comparative counts of THB and THU Bin soil from day0-35 across all microcosms

treatments maintained similar trend throughout the experiment. One way ANOVA revealed no Significant difference (SD) ( $P < 0.05$ ) observed between treatments at day 35 except casmes and NPK (0.049), coco dust and NPK (0.014).

For THUB, CM, CM+CD, CCD and OSM microcosms showed similar growth pattern from day 0 to 35. THUB counts reduced by an order of magnitude ( $10^7$  cfu/g) at day 7 with respect to the initial THUB population at day 0 ( $10^8$  cfu/g), increased on day 14, and gradually declined till day 35. For NPK microcosm, counts increased on day 14 slightly, retaining same order of magnitude with day 7 ( $10^8$  cfu/g) and gradually decreased till day 35. Coco dust microcosm had the maximum THUB counts of  $3.43 \times 10^7$  at the end of the experimental period and this was closely followed by osmocote ( $2.61 \times 10^7$  cfu/g) while the least THUB count was observed in CM+CD ( $8.97 \times 10^6$  cfu/g). The results suggests

that the formulated SRFs effectively supplied the indigenous bacterial population with nutrients to sustain their growth even in a stressed environment as evidenced in the result of the bacterial growth profile. Interestingly, the super absorbent polymer coat upon completion of the nutrient release process, completely degrades enriching the soil and consequently the microbial community [19]. One of the known and most studied slow release fertilizers is osmocote which has been widely used in laboratories and mesocosm bioremediation studies have shown to enhance and facilitate the growth and activities of oil degrading microbes [20, 21]. In a related study by Darmayati and Afianti [22] The local slow release fertilizers produced (MLT, Grand TMM) showed favourable results as candidates for growth stimulation of microorganisms during bioremediation studies. The study revealed that the three locally formulated SRFs were able to stimulate indigenous bacteria growth more than Osmocote.

#### 4. CONCLUSION

This study highlighted the successful formulation of two slow release fertilizers (cassava and coco dust) from natural based polymers as sustained nutrient delivery supplements. The low cost crop residues (cassava mesocarp and coconut coir dust) modified with gelatinized cassava starch as binding agent were adequate in the successful formulation of 100% natural based super absorbent polymers (CM-SAP and CCD-SAP) applied in producing two slow release fertilizers that can be used in both agriculture and enhancing environmentally beneficial soil microorganisms for bioremediation.

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#### COMPETING INTERESTS

The authors declare that there are no conflicts of interest.

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