Research article

Effect of harvesting stage, form and duration of storage on some physico-chemical properties of Korarima (*Afframomum* (Braun) P.C.M. Jansen) collected from Masha Woreda, Sheka Zone, Southwestern, Ethiopia

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**ABSTRACT**

Korarima, also called Ethiopian cardamom, is cultivated in the forest areas of southwestern part of Ethiopia. It is an underutilized spice crop with minimal research attention in the past. Therefore, a study was carry out to determine the appropriate harvesting stage, form, and duration of storage for an optimum quality of korrorima. The experiment consists of two different harvesting stages (fully mature green and mature red), three forms of storage (capsule, seed and powder) and three storage durations (0/without storing, 3 months storage and 6 months storage time) were arranged in factorial combination in randomized complete block design with three replicates. Results indicated that Fresh weight of capsules (FWC), dry weight of seeds (DWS), and seed to husk ratio (STHR) were highly significantly affected by harvesting stages (*P* < 0.01). Significant (*P* < 0.05) to very highly significant (*P* < 0.001) variation was also observed for seed quality traits such as volatile oil and oleoresin contents. The mean values of the fresh weight of capsule for mature green were 24.55 g and 23.28 g for mature red; dry weight of seed for mature green were 4.59 g and 6.45 g for mature red; and seed to husk ratio for mature green was 2.65 g and 3.38 g for mature red. Whereas, the available moisture content per sample ranges 15.08%–17.86%; crude protein 7.43%–9.39%; Volatile oil content ranged from 2.50 (v/w %) to 4.80 (v/w %) and oleoresin from 5.06 (v/w %) to 9.93 (w/w %). The variation may be linked to the moisture content and accumulation of different assimilates. Furthermore, level of exposure to higher temperature and free reactive oxygen might have created a significant variation between samples during the storage time. Fully mature green stage harvesting was found more recommendable and acceptable but fails to retain its intrinsic quality during long storage. Prolonged harvesting reduces most of the intrinsic quality of the A.corrarima. Storing for extended time in the form of powder may lead consumers to loss most quality traits than storing in the form of capsule.

**1. Introduction**

A spice is a dried part of vegetative substance which are seed, fruit, root or bark used in nutritionally insignificant quantities as a food additive for the purpose of flavoring, and sometimes act as an antibacterial by killing or avoiding the growth of harmful bacteria (Masresha, 2010). According to Herms (2015), spices are one of the traditional high value horticultural crops of Ethiopia. Most spices originate in tropical and semi-tropical developing countries (Raghavan, 2007) due to this, production of most spices and herbs have been traditionally concentrated in these tropical regions of the world. Even though the sights have been highly productive, sanitation and food handling practices may not be adequate since most of the small-scale producing farmers may not be fully aware of the need to protect their spice crops from conditions that lead to the presence and growth of pathogenic microorganisms which affect quality of the products (ASTA, 2011).

Most indigenous African spices like *Aframomummelegueta, Aframomumcitratum, Piper guineense and Xylopiaaethiopica* are often advantageously used as low cost eco-friendly, low risk pesticide alternatives to the conventional synthetic pesticides (Ntonifor, 2011). *Afframomum* (Braun) P.C.M. Jansen is one of the most widely used spices in Ethiopia. It is used to flavour food and beverages in Ethiopia (Hymete et al., 2005). The spice, Ethiopian cardamom (Jansen, 1981, 2002) is obtained from the plant’s seeds (usually dried), and is extensively used in Ethiopia and Eritrea cuisine. It is ingredient in most of food mixture like -berbere, [[mitmita] ,-jawaze and is also used to flavor coffee. In Ethiopia herbal
2. Materials and methods

2.1. Description of the study area

Corrorima was collected from the forest of Masha Woreda, Sheka Zone, South Nation Nationalities and Peoples (SNNP) regional state in the year 2018/2019. Geographically, the Zone lies between 7°24’ and 7°52’ N latitude and 35°13’–35°35’ E longitude with Zonal cover of about 2175.25 km2, out of which, 47% is covered by forest, including bamboo. Masha Woreda is located 672km away from Addis Ababa. The altitudinal range of the areas in the Zone falls between 900 and 2700 meters above sea level. The cultivation practice in small holders ‘farm’ is fragmented and planted as mixed crop within their main crop land and rain fed (Spice Sector Strategy Coordinating Committee, 2010).

A problem, especially with forest *A. corrarima* is that people have to walk long distances to find enough fruits and difference in stages of maturity on the fruits. In most cases it could be too much work to come back after some days to collect the fruit and people will instead collect all fruits regardless of the maturity stage. Unfortunately, these harvests were pass a variety of post-harvest challenges and reach to different level of consumers and retailers. It is obvious that the production is season based and the consumption is all year round. To make the spice available and consume for extended period of time people store it in different forms for different storage durations.

Standing from previous trends in most of southwestern Ethiopia, mainly *korrorima* producing areas, the price of the commodity is season based; it is not either cost or demand based. Due to this reason, many collectors prefer to collect during production season, and store the spice for short to long period of time for seeking more market opportunity. The production of this spice is continuing and the demand for the spice is also increasing. In addition to this, researches indicates that the crop has a capacity to compete, even replace cardamom but still its overall quality is facing a huge problem due to lack of knowledge about harvesting stage, form and duration of storage. It was this factor that inspired for the study in order to investigate the problems and prospects of these indigenous spices. The present study was, therefore, Determine the appropriate harvesting stage, form, and duration of storage for an optimum quality of *Korarima*.

2.3. Laboratory analysis

2.3.1. Physical characteristics

The physical parameters such as fresh weight of single capsule, dry weight of seeds per capsule and seed to husk ratio were taken.

2.3.2. Moisture content (proximate analysis)

The moisture content for the samples were determined by using the method of AOAC (1990).

2.3.3. Hydro-distillation of essential oils (w/v)

The essential oil isolation from was carried out according to ASTA (1997) procedure. The amount of volatile oil was determined by modified Clevenger method. Hundred gram of ground sample was weighed and transferred into a 500 ml round bottom flask. One litter of distilled water was added to the sample containing flask. The trap was filled with water. The condenser was connected to the system and the whole system was fixed onto a stand on heating mantle. The flask was heated to boil, and the distillation was continued until two consecutive readings taken at 1hour intervals showed no change in oil content. It took about 3–4 h to complete each cycle of distillation process. Finally, the source of heat was removed and the volume of oil was recorded. Then the extracted oils were carefully separated from its hydrolytes. Each oil sample was allowed to dry by putting anhydrous sodium sulphate (Na2SO4) and then pure oils were harvested.

2.3.4. Acetone extractable solutes (oleoresin)

In this method, the ground sample was placed inside a thimble made from thick filter paper. The thimble was placed in an extraction chamber which is suspended above a flask containing the solvent and below a condenser. The flask was heated to evaporate solvents to move up into the condenser where solvent vapors converted to a liquid that trickles into the extraction chamber containing the sample. After many cycles the desired compound was concentrated in the distillation flask, the process the extract was transferred into a beaker quantitatively. The complete evaporation of the solvent was done by using a steam bath (Heidolph, rotary evaporator, Germany) apparatus. When the last traces of acetone

| Maturity stage | FWC(kg) | DWS(kg) | STHR (gm) |
|----------------|---------|---------|-----------|
| Mature green   | 24.55±  | 3.53±   | 2.65±     |
| Mature red     | 23.28±  | 6.45±   | 3.38±     |
| LSD            | 0.327   | 0.163   | 0.074     |
| SE (±)         | 0.16    | 0.29    | 0.13      |
| Cv (%)         | 2.5     | 5.3     | 4.4       |

FWC-fresh weight of capsule; DWS- dry weight of seed; STHR-seed to husk ratio; gm-gram.

Means sharing the same letter(s) in the column are not significantly different at p = 0.05 according to LSD test.
molecules were evaporated, the container was placed in a hot air oven at 110° ± 2°C until two consecutive weightings taken at 11/2-hour intervals didn’t differ by more than 1mg. The dried residue was the non-volatile oleoresin (ASTA, 1997).

2.4. Data collected and analysis

To determine the appropriate harvesting stage, form, and duration of storage for an optimum quality of data were collected for physical parameters such as: Fresh weight of single capsule (g), Dry weight of seeds per capsule(g), Seed to husk ratio; and Phytochemical analysis such as: Seed moisture content determination; Hydro-distillation of essential oils, and Acetone extractable solutes (oleoresin) content determinations are the other quality characters which are measured for evaluating the treatments.

2.5. Statistical analysis

All the collected data were first checked for fitting the analysis of variance (ANOVA) assumptions. Then, all data were subjected to analysis of variance (ANOVA) using SAS 9.3 version (SAS, 2012). Means were compared by using least significant difference (LSD) test at 5% probability level. The significant difference between the treatment mean were separated by using a letters from a to z. Homogeneity of the variance (Bartlett’s test) was tested by using Minitab 15 (Minitab version 15, Minitab Inc., State College, PA, USA) statistical software to check the validity of the data and transformation of data was carried out for those who failed the test. The presentation of the mean values was done after retransformation of the transformed data’s were carried out.

3. Results

3.1. Characteristics of physical parameters

The physical parameters considered in this study were Fresh weight of single capsule, dry weight of seed and seed to husk ratio. ANOVA results and the means are shown in Table 1 below:

3.1.1. Fresh weight of single capsule

The mean values for fresh weight of the samples showed a very highly significant difference (p < 0.01) between the two harvesting stages of A. corrorima (Table 1).

The maximum value of average weight of single fresh capsule was observed at mature green stage (24.6g) followed by mature red stage (23.3g) capsule (Table 2). The average fresh weight of capsule in these study ranges from 22.07g to 25.5g and with the grand mean of 23.91g. This is may be due to the accumulation of higher amount of moisture during the early maturity stage of the capsule than the dry matter. Fully ripe and mature fruits should only be harvested once they are ready.

3.1.2. Dry weight of seed (g)

The analysis of variance on dry weight of seed (DWS) shows a very highly significant difference (p < 0.001) among the two maturity stage harvested A. corrorima (Table 1). The highest dry weight of seed per capsule was recorded at mature red stage harvest (6.45g) and followed by mature green (3.53g) stage harvested A. corrorima. This may be indicating that when ripening begins following the physiological maturity, metabolic activity will take place which may result in increasing of the dry matter content and reduction of moisture contents inside mature red stage seed.

3.1.3. Seed to husk ratio

The analysis of variance shows a very highly significant difference (p < 0.01) between the two maturity stages of harvested in terms of seed to husk ratio (Table 1). The maximum STHR was observed on mature red stage harvested (3.4:1) and followed by mature green stage harvested (2.6:1) (Table 2). These may indicate that during maturity, most of the nutrients can be allocated to the seed than the husk as a result, the seed constitute during maturation increase and the capsule become expand and reduces its thickness, finally after full maturity of the seed, the husk of the seed become low in thickness and loses most of its moisture content with in a cell.

3.2. Characteristics of chemical parameters

Moisture contents in all treatment samples were determined by using standard procedures prescribed by AOAC (1990) and ASTA (1997). The means of different maturity stage, form of storage for collected sample were tested at zero, three and six month’s storage time. Both types of maturity stage at different form of storage results were statistically analysis and presented in Tables and discussions.

3.2.1. Moisture content (%)

Moisture is one of most important factors affecting seed quality from the time seed mature in the field until they are planted. Moisture determines how long mature seed will maintain high quality.

The ANOVA result indicates that there was a very highly significant difference (p < 0.001) on the moisture content due to the interaction effect of the three factors. The sample which was harvested at mature green stage and stored in the form of whole capsule for zero, three- and six-months storage time recorded highest moisture contents which is 16.70%, 16.25% and 15.87%, respectively. While, mature green stage harvested korarima and stored in the form of seed for zero months storage period exhibit 13.40% and followed by three months stored (12.74%) and the least was recorded at six months (12.39%) storage period. At the same maturity stage, the sample stored in the form of powder for zero-, three- and six-month storage period records 12.59%, 12.15% and 11.41%, respectively and statistically similar with that of mature green stage harvested and stored in the form of seed for six months of storage time.

During this time the moisture content of the seeds may start to decline gradually as the dry mass increases. In the early stages of development immediately following fertilization, the fresh or dry mass of the seed is low and the moisture content high (Eskandari, 2012). This observation is consistent with previous study which was (25.6g) for mature green and (22.52g) of fresh weight of red A. corrorima capsules (Fissiha, 2012).

This may indicate that initially the moisture content of mature green stage A. corrorima has higher and experienced the highest moisture loss
throughout storage duration. When highest moisture content was supported by grinding (i.e. increasing of surface area), higher temperature and storage period leads the sample to loss highest moisture than the one which is at mature red stage harvested corrorima. This may show that higher exposure due to grinding in powdered form expected more loss than that of other form stored korrorima samples. It is well known that form at which the spices stored may determine the life spine of the commodities, so as the current study also strengthen the above idea. As result the moisture loss observed on sample stored in the form of whole capsule records 1.06%, followed by seed 1.17% and the highest is recorded in powdered form stored sample which is 5.05%. In seed form stored sample comparatively the surface area is lower than the powder form and maintains and provides most of the internal contents including moisture and flavor at a more economical price. This is may be due to the waxy layer of the seed cote which may prevent the out flow of moisture partially.

Comparative with the current finding Fissiha (2012) reported that the moisture content for mature green and mature red capsule korrorima consists of 16.04% and 15.22%, respectively. Similarly, Chempakam and Sindhu (2008) studied physicochemical qualities of large and small cardamom seeds and identified 8.49% and 8.30% moisture content for large and small cardamom, respectively. Researches done by Rizhakkayil and Sasikumar, 2009 to compare physical and biochemical quality of Indian, Guatemalan and Sri Lankan cardamoms indicates that the moisture content for the three cardamoms are 5.08, 15.74 and 18.84, respectively. Hence, our current result was superior to the former two cardamoms but inferior to that of Sri Lankan (18.84).

3.2.2. Hydro-distillation of essential oils (v/w %)

The ANOVA result indicates that there is a significant difference (p < 0.001) on the essential oil content of A.corrorima (Table 3). A sample which was collected at mature green stage and stored in the form of whole capsule for zero storage months consists of the highest oil content 4.3v/w%, followed by seed form stored sample for zero months 4.2v/w% which was statistically similar with that of mature red harvested and stored in the form of whole capsule for the same storage period 4.2v/w%, whereas, mature green stage harvested and stored in the form of whole capsule for three and six months stored sample obtain 3.9v/w%, and statistically similar with that of mature red stage harvested and stored in the form of whole capsule for three months 3.9v/w% and six months of storage time 3.9v/w%. The least oil content was observed on mature green stage harvested and stored in the form of powder for six months of storage periods 2.8v/w %.

As evident from the result (Table 3), there was a decline of oil content by 2.3v/w % for the mature green and 1.4v/w % for the mature red harvested A.corrorima. This indicates that comparatively faster oil degradation was observed in mature green than mature red. This is may be because of one of the following factors, one is due to less level of fiber in mature green harvested than the mature red.

3.2.3. Acetone extractable solutes (oleoresin w/w %)

Spice oleoresins represent the complete flavour profile of the spice. Spices are well-known by their volatile as well as nonvolatile constitu-ents. The interaction effect of maturity, form with duration on oleoresin content of the samples show a very highly significant variation (p < 0.001) (Table 4).

A sample which was collected at mature green stage and stored in the form of capsule for zero-month storage consists of the highest oleoresin content 7.95w/w, followed by mature red stage harvested and stored in the form of capsule for three months of storage time records 7.82w/w which is statistically similar with mature green stage harvested and stored in the form of seed for zero months 7.66w/w of storage time. Mature green stage harvested and stored in the form of whole capsule for three months of storage records 7.56w/w which is statistically comparable with mature red stage harvested and stored in the form of seed for zero month7.52w/w, whole capsule for zero month 7.50w/w storage.

4. Discussion

To get better quality A.corrorima, the fruits should only be harvested once they are fully ripe and mature. During this time the moisture content of the seeds may start to decline gradually as the dry mass increases. In the early stages of development immediately following fertilization, the fresh or dry mass of the seed is low and the moisture content high (Eskandari, 2012). This observation is consistent with previous study which was (25.6g) for mature green and (22.52g) of fresh weight of red korrorima capsule (Fissiha, 2012).

In our current study, both maturity level seeds of A. corrorima were found to comprise 70.07%-78.93% of the matured and dried whole fruits; the rest was due to the husks which ranges from 21.07%-28.93%. This difference may also relate to the amount of nutrient and moisture accumulation taken from the soil. Principally, accumulation of most nutrients was highest in vegetative growth parts of the plant at pre- bearing stage and at harvest stages these allocations of nutrient turns to seed capsule formation and records the highest values. Growth environ- ment play profound impact on seed quality of most crops, mainly because of their impact on nutritional uptake, assimilate supply, partitioning and remobilization of nutrients (Prasad et al., 2008). In most seed-bearing spices when they attain their physiological maturity there is

| Maturity stage | Form       | Duration (months) | 0     | 3     | 6     |
|----------------|------------|-------------------|-------|-------|-------|
| Mature green   | Whole capsule | 4.3a               | 3.9bc | 3.9bc |
|                | Seed        | 4.2b               | 3.6de | 3.4ef |
|                | Powder      | 3.9bc              | 3.8ef | 2.8f  |
| Mature Red     | Whole capsule | 4.2b               | 3.9bc | 3.8ef |
|                | Seed        | 3.9bc              | 3.4ef | 3.1f  |
|                | Powder      | 3.4ef              | 3.1f  | 3.2fg |

Means sharing the same letter(s) are not significantly different at p = 0.05 according to LSD test.

| Maturity stage | Form       | Duration (months) | 0     | 3     | 6     |
|----------------|------------|-------------------|-------|-------|-------|
| Mature green   | Whole capsule | 7.95a              | 7.56bc| 6.47f |
|                | Seed        | 7.66bc             | 6.89bc| 6.66df|
|                | Powder      | 6.58df             | 5.82f | 5.55f |
| Mature Red     | Whole capsule | 7.50bc             | 7.82bc| 7.22df|
|                | Seed        | 7.52bc             | 7.50bc| 6.50df|
|                | Powder      | 6.52df             | 5.13f | 5.00f |

Means sharing the same letter(s) are not significantly different at p = 0.05 according to LSD test.
a rapid increase in size and weight (Ekpong and Sukprakarn, 2008) and after physiological maturity the moisture contents become lower, the capsule of the fruit starts to turn its colour and the dry matter reaches its maximum dry weight. At this point the seed could already have a capacity to germinate and the fruit is botanically mature (Biglari, 2009).

The seed dry weight obtained in this study was inconsistent related to maturity level difference with the work of Fissiha (2012) who reported that dry weight of seed content was 6.65g for mature green and 6.32g for the mature red A. corramina but it is consistent with dry weight content of seed percentage range. Gurpınar and Mordogan (2005) who reported the variations in seed dry weight among maturity stage may be due to the amount of nutrient taken from the soil, which was proportional to the nutrients present in soil.

In a previous study, which was done by Fissiha (2012) reported that 2.42:1 to 3.36:1 ratio of seed over husk were obtained, and slight difference was observed with current study. The other author Amma et al. (2010) reported on his study of physico-chemical qualities of four major varieties of cardamom viz. Mysore, Malabar, Vazhukka and Guatemala from India and found seed to husk ratio of 62:38, 76:24, 70:30 and 66:34, respectively which was by far lower than the present findings. Study on physical and biochemical quality parameters of Indian, Guatemalan and Sri Lankan cardamoms indicates that the seed to husk ratio of 3:1, 1.7:1 and 2:1:1, respectively (Kizhakkayil and Sasikumar, 2009).

The other could be the level of exposure due to grinding may create a suitable condition for oxidation by increasing the surface area. The size reduction by grinding and exposure of the internal constituent to the environment may create a favorable condition for reactive free oxygen; in addition to these the low fiber content in mature green powdered form of the sample may also determine the frequency of the degradation. As a result, oxidation and degradation of Essential oil was higher in mature green stage harvested and stored in the form of powder 1.10v/w% than the seed form stored sample in both maturity stage 0.80v/w% and the least was observed on capsule form stored samples both in mature green and mature red stage harvested 0.4v/w%.

Grinded spice deteriorates in its aroma quality both by rapid loss of volatile and by the action of oxygen in the head space on the terpenic and lipid components of the spice (Peter, 2001). In pepper the volatile oil percentage (%) decreases with berry maturity but piperine (%) and starch % increase (UNIDO and FAO, 2005). Essential oils are known to be susceptible to conversion and degradation reactions. Quality loss of spices can be strongly supported by Oxidative and polymerization processes. To a decisive degree, its degradation depends on several chemical and edaphic factors that influence both the possibility of the essential oil to oxidize as well as the course of the reaction. Therefore, a significant considerations should be required for external factors such as temperature, light, and accessibility to atmospheric oxygen need. Furthermore, essential oil composition, compound structures, and the presence of impurities may also govern stability (Turek and Sintzing, 2013).

At high storage temperature naturally existing essential oil components in most of the spices are subject to oxidation by atmospheric oxygen, particularly which resulting in the development of off-flavours (Peter, 2006). Due to their structural relationship within the same chemical group, essential oil components are known to easily convert as result it is recommended to store in bottle with tap (air tight bottle) and low ambient temperature. Hence, storing A. corramina for up to six months with the capsule may don’t have create significant quality deteriorations but it is better to consume seed and powder form before three months storage time is more advisable.

lower than that of Amma et al. (2010) which has done on four major E. cardamom varieties (Mysore, Malabar, Vazhukka, and Guatemala) have reported as 7.9, 8.79, 7.9 and 8.6, respectively. A study on different ginger varieties indicates the percentage contents of essential oils and oleoresin decreased with increasing maturity (Ravindran and Babu, 2005). Eyob et al. (2007) reported the essential oil yield from dried seeds of highland 4.30% oil yield and was higher when extracted from fresh samples compared to dried seeds 3.77%. However, the volatile oil content of the samples in both maturity stage was found to be much lower than from Indian (10%), Guatemalan (5%) and Sri Lankan (14%) cardamom (Thomas et al., 2006) as well as from Indian corramina of different varieties (Amma et al., 2010).

The percentage composition of essential oils and oleoresins can be decrease with increasing of maturity (Parthasarathy et al., 2008). There are various factors determine the chemical composition of cardamom which are variety, region and age of the products are considerably the affecting factors. The content of volatile in the seeds is strongly dependent on storage conditions (Parthasarathy et al., 2008). The gingersols are susceptible to chemical transformation to less pungent degradation products and that these reactions can occur by poor handling during the preparation, storage and use of dried ginger and its oleoresin, with consequent deterioration of quality (Parthasarathy et al., 2008). When ginger stored at room temperature (23–26 °C), losses of up to 20% oleoresin (dry weight) were observed on dry ginger after 3 months, and the content of (6)-gingerol decreased (FAO, 2002).

These observations may be suggested that decline of seed oleoresin content was examined in case of maturity stage, 3.51w/w 15.8% for mature green and 2.76w/w 12.81% for mature red not only that, but also in case of form of storage there is high variation existed among the three form of storage 1.76 w/w 11.39%, 1.96w/w 12.91% and 2.55w/w 19.5% for capsule, seed and powder form, respectively. This indicates that when the level of maturity increases the percentage composition of oleoresin content will be decrease. The decrease in oleoresin contents of with advancing maturity may be attributed to continued physiological activity for color change and in response to external stimuli using from these compounds as precursors. The result obtained on this study was in line with the previous reports (Fissiha, 2012) for oleoresin content at its maximum record which is 4.87–9.16w/w. Other studies on small cardamom grown in different regions of India shows non-volatile ether extracts (%) ranging from 2.0-3.6% (Chempakam and Sindhu, 2008).

5. Conclusion

The quality obtained from fully matured green stage harvested A. corramina can exhibit the highest quality profile in most quality determinant factors; hence, harvesting koraima at fully mature green stage is more recommendable for fresh consumption and for source of secondary metabolites (mature green oil 4.13v/v oil, 7.39%w w oleoresin). In contrast, mature green harvested koraima has low dry mater accumulation than that of mature red stage harvested koraima as result it is loss for dry matter-based consumers, not only that but also there is a great loss of internal constitute during extended storage periods in mature green stage harvested koraimina than that of mature red stage harvested koraimina.

Prolonged harvesting reduces most of the intrinsic quality of the A.corramina flavor can be readily oxidized, and losses occur during desiccation and milling, as a result storing seed and powder form for extended time cannot be recommendable unless they are stored in bottle with tap (air tight bottle) and low ambient temperature. Hence, storing A.corramina for up to six months with the capsule may don’t have create significant quality deteriorations but it is better to consume seed and powder form before three months storage time is more advisable.
Declarations

Author contribution statement

Abeaw Hailegeorgis: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Daniel Anbesse: Performed the experiments; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data included in article supplementary material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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