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Optimising Process of Natural Dyeing of Cotton with Annatto Seeds (*Bixa orellana*) Using Natural Mordants

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ABSTRACT

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The research focused on optimizing the process of natural dyeing of cotton using Annatto seeds (*Bixa orellana*), a vibrant yellow-orange dye derived from plant sources. The study aimed to enhance the sustainability and color fastness of cotton textiles by exploring various factors such as dye extraction time, dye amount, dyeing time, mordanting techniques, and mordant types. Natural mordants including alum, aloe vera gel, lemon juice, tamarind leaves, and harda powder were selected for their eco-friendly properties. The study revealed several key findings. First, the optimal dye extraction time for Annatto seeds was found to be 15 minutes, yielding the highest absorption rate. Similarly, the most efficient dye amount was 15 grams, and the ideal dyeing time was 45 minutes. Among the natural mordants, alum (4 grams) was most effective, followed by tamarind leaves (2 grams), which contributed to the brightest colors and best dye absorption. Pre-mordanting methods were more effective than post-mordanting, especially when using tamarind leaves and harda, which showed the highest dye absorption rates. The results emphasize the importance of optimizing the dyeing process to improve resource efficiency and enhance the ecological sustainability of textile production. The optimized conditions proposed in this study—using 15 grams of Annatto dye, 4 grams of alum, and dyeing for 45 minutes—can serve as a guideline for more sustainable cotton dyeing practices in the textile industry, reducing both material waste and environmental impact.

Introduction

The textile industry has long relied on synthetic dyes for coloration, but growing environmental concerns have spurred a resurgence in the use of natural dyes derived from plant, animal, and mineral sources (Adeel *et al.*, 2021). Among these, natural dyes from plant materials have gained attention for their eco-friendly nature, biodegradable properties, and reduced environmental impact. One such plant-based dye is Annatto (*Bixa*

orellana), a vibrant yellow-orange dye extracted from the seeds of the Annatto plant. This dye has been traditionally used in a variety of applications, from food coloring to textile dyeing, due to its bright and long-lasting hues (Fursule, 2004; Amutha, *et al.*, 2020).

Cotton, a widely used natural fiber, presents unique challenges and opportunities in the dyeing process. The inherent qualities of cotton—its cellulose-based structure—make it less receptive to natural dyes

compared to protein-based fibers like wool or silk (Sinnur, 2018; Shenai, 2000). To overcome this limitation, the use of mordants, substances that fix the dye to the fabric, is essential. Traditional mordants such as alum, iron, and tannins can enhance the dye's uptake and color fastness, but their application needs careful optimization to maximize efficiency and minimize environmental harm (Hasan, 2015; Jahan, 2015; Krizova, 2015).

The process of natural dyeing with *Annatto* seeds, when combined with various natural mordants, offers a promising solution for producing eco-friendly, vibrant cotton textiles. However, there is still much to learn about optimizing the dyeing process to improve color intensity, fastness, and overall textile quality while reducing resource consumption. This research aims to explore and optimize the process of natural dyeing of cotton using *Annatto* seeds (*Bixa orellana*) in conjunction with natural mordants (Kumar, 2021; Kumar, 2023).

By investigating different mordanting techniques, dyeing conditions, and their interactions, the study seeks to develop a more efficient and sustainable method for producing high-quality, color-fast cotton textiles. The findings of this study could contribute to the growing field of sustainable textile production and promote the use of natural resources in the fashion and textile industries.

Materials and Methods

Selection of substrate

100% cotton was selected for the study as it is easily available and widely used natural fiber. Some technical and non-technical tests were done for the confirmation of purity of cotton. Solubility test with 70% H₂SO₄ and microscopic test for longitudinal appearance was done as technical test and for non-technical test, burning test was done.

Pretreatment of substrate

The cotton fabric was scoured. It was soaked in warm water overnight which helped to remove the finishing chemicals applied to it. It helps dye to penetrate and dye evenly.

Selection of Natural dye and natural mordants

Natural Dye *Annatto* seeds (*Bixa orellana*) was selected for present study. Five natural mordants were used in the present research work. Alum crushed into powder, Aloe Vera gel extracted from fresh leaves, lemon juice, fresh tamarind leaves and whole harda powder were the mordants used.

Selection of Medium of dye extraction

Annatto seeds were extracted in 100 ml of water and 10gm of dye in each acidic, neutral and basic mediums. For acidic medium few drops of Sulphuric acid was used. For basic medium a pinch of Sodium carbonate anhydrous was used. The medium which gave bright and dark colours was selected on the basis of visual comparison for further study

Optimization of dye extraction time

For optimization of dye extraction time 10 gm of dye stuff was boiled in 200 ml of water in basic medium for 6 different time duration i.e. 15 min, 30 min, 45 min, 60 min, 75 min and 90 min each. The beakers were sealed for no water loss. Digital visible spectrophotometer was used to get the Optical density (OD) of the extractions at different time duration and results were noted. For dyeing 6×6 inches' cotton fabric samples were dyed in dye solution for 30 mints at 100⁰ C which is extracted for different time durations. Beakers were sealed so that no amount of water evaporates. OD of dye before and after dyeing were noted. Percentage of absorption of was calculated. Time with highest percentage of absorption was selected as extraction time for all further processes.

$$\% \text{ of absorption} = \frac{\text{OD before dyeing} - \text{OD after dyeing} \times 100}{\text{OD before dyeing}}$$

Optimization of dye amount

For dye amount 10gm, 15gm, 20gm, 25gm and 30gm of dye was extracted in 200 ml of water in basic medium separately. Dye was extracted for selected extraction time and samples were dyed for 30 mints. All beakers sealed so that water does not evaporate. Optical density was noted and percentage of absorption was calculated and dye amount with highest percentage of absorption was selected as dye amount for all further processes.

Optimization of dyeing time

For selection of dyeing time selected amount of dye stuff was extracted in 200 ml of water in basic medium for selected extraction time. Dyeing was done for 15 min, 30 min, 45 min, 60 min, 75 min and 90 min each. OD was noted before and after dyeing and percentage of absorption was calculated and time with highest percentage was selected for all further processes.

Optimization of mordant amount and mordanting methods for dyeing of cotton with Onion skins

Optimization of mordant amount

Simultaneous mordanting method was used for the optimization of mordant amount. Dye was extracted in standard conditions and simultaneous mordanting was done with 2 gm, 4 gm, 6gm, 8 gm, 10 gm and 12 gm amount of all five mordants i.e. Alum crushed into powder, Aloe Vera gel extracted from fresh leaves, lemon juice, fresh tamarind leaves and whole harda powder. OD was calculated before and after dyeing. Percentage of absorption was calculated and amount of mordant with highest dye absorption was finalized.

Mordanting method

For pre mordanting, mordant solution was prepared as per the optimized amount and samples were mordanted for 30 min. After mordanting samples were dyed according to the optimized dyeing process. For post mordanting, samples were dyed according to the optimized process and mordanted in optimized amount of mordant solution for 30 min. OD was calculated before and after dyeing, and best mordanting method was finalized.

Results and Discussion

Confirmatory test of Cotton

For solubility test small piece of cotton substrate for treated with 70% H₂SO₄ and cotton dissolved in it. In microscopic test longitudinal appearance was observed and fibers looked flat and twisted. In burning test, a small piece of fabric was burnt to check the purity for 100% cotton poplin. It ignited quickly and burnt fast. It smelt like burnt paper and flaky grey ash was left behind. It all concluded that substrate was 100% pure cotton.

Optimization of dye extraction time

10 gm of annatto seeds dye was extracted in 200 ml of water in basic medium for 15 min, 30 min, 45 min, 60 min, 75 min and 90 min each. Samples were dyed in the extract for 30 min. Following are the results for the same.

It is evident from the results that; the highest percentage of absorption was for 15 min of dye extraction both for annatto seed dye. So 15 min was optimized as dye extraction time for all further processes. Results shows that extraction for long time do not yield more dye, hence resources can be saved by extracting dye to the minimum time required.

Optimization of dye amount

10 gm, 15 gm, 20 gm, 25 gm and 30 gm of Annatto seed dye was extracted for optimized extraction time i.e. 15 min of annatto in 200 ml of water in basic medium. Samples were dyed for 30 min in the extract made by optimized process and results are tabulated as follows.

On the basis of above results in Table-3, the highest percentage of absorption was for 15 gm for annatto seeds. The above tabulated results show that more dye concentration does not help the substrate to absorb more, so minimum required amount should be used and raw material should be used without wasting.

Optimization of dyeing time

As per optimized process till now 15 gm of annatto seeds were extracted for 15 mins each in 200 ml water in basic medium. Then samples were dyed for different time durations from 15 min, 30 min, 45 min, 60 min, 75 min and 90 min and percentage of absorption was calculated. Following are the results for the same.

On the basis of above tabulated results final optimized recipe for dyeing comes out as:

Optimized Dyeing Conditions for Annatto seeds dye on Cotton

Medium of dye extraction: Basic

Amount of Liquor: 200 ml

Dye extraction time: 15 min.

Dye amount :15 gm.

Dyeing time: 45 min.

Optimization of Mordant amount

For optimization of mordant amount simultaneous mordanting method was used. Optimized recipe of dyeing was used and with it 2 gm, 4gm, 6 gm, 8 gm, 10 gm and 12 gm of mordants each for annatto seeds were used. Following are the results of percentage of absorption of the same.

Optimization of amount of Alum powder

From the above results it is clear that 4 gm of alum (Potassium aluminium sulphate – $K_2(SO_4) \cdot Al_2(SO_4)_3 \cdot 24H_2O$) or also known as Aluminium hydroxide gel in medicine, 'Turti' in Marathi and 'Fitkari' in Hindi in annatto seeds were optimized amount of alum mordant. Alum lightens the colour shade of annatto seeds dye and it gives a patchy crepe effect to the fabric.

Optimization of amount of Aloe Vera gel

From the above results it is concluded that 12 gm Aloe Vera (*Aloe vera*) also known as 'Korfad' in Marathi and 'Ghrut Kumari' in Hindi is best amount for optimized dyeing recipe of annatto seeds dye.

Amount of Aloe Vera needed is quite high as compared to other mordants used in study. Aloe Vera as mordant makes the cotton fabric soft and smooth in feel. It also fades the shade a bit. For annatto seeds the shade does not get affected much.

Optimization of amount of Lemon juice

From the above tabulated results, it shows that 4 gm of lemon juice is required for optimized dyeing recipe of annatto seeds. Lemon (*Citrus limon*) also known as 'Limbu' in Marathi and 'Nimbu' in Hindi as mordant gives a citrusy fresh fragrance to the fabric. Colour shade fades for annatto dye.

Optimization of amount of Tamarind leaves

From the above results it is revealed that 2 gm Tamarind leaves (*Tamarindus indica*) also known as 'Chincha' in

Marathi and 'Imli' in Hindi in optimized recipe of annatto seeds dye gives best absorption. Tamarind leaves as mordant gives bright colour and colour shades do not change, remains like the optimised recipe dye colour shade. Tangy aroma is left in the substrate which is quite refreshing.

Optimization of amount of Harda powder

From the results it is clear that 2 gm of harda (*Terminalia chebula*) also known as 'Myrobalan' in English and 'Harad' in Hindi is optimised amount of mordant both for annatto seeds. Samples dyed in harda has slightly less colour shade than that of optimised dyeing sample. Samples also has some deposits of harda powder.

Optimization of Mordanting methods

All the above mordanted samples were done by simultaneous mordanting. Next mordanting was done with all five selected mordants with per mordanting and post mordanting.

For post mordanting samples were dyed with optimized recipe and then mordanting was done in optimized mordant amount. OD of dye for post mordanting was not done as it gives result only for dyeing process and not for mordanting. Following are the results for pre mordanting. In pre mordanting colour shades do not change for annatto seeds dye. But in post mordanting annatto dye shared changes a bit towards pinkish instead of bright orange.

The research demonstrates the potential of Annatto (*Bixa orellana*) as an eco-friendly and effective natural dye for cotton textiles. Through careful optimization of the dyeing process, including dye extraction time, dye amount, dyeing time, and mordant selection, the study successfully identified conditions that maximize the dye's absorption and color fastness while minimizing resource use.

The optimized process for dyeing cotton with Annatto seeds involved extracting the dye in a basic medium for 15 minutes, using 15 grams of dye, and dyeing the fabric for 45 minutes. These conditions ensured the highest percentage of absorption, promoting efficient use of resources.

Table.1 Selected natural mordants for the study

Sr. No.	Mordant name	Botanical name	Hindi name	Marathi name
1.	Crushed alum powder	Potassium aluminium sulphate	Fitkari	Turti
2.	Fresh aloe vera gel	<i>Aloe vera</i>	Ghrut Kumari	Korfad
3.	Lemon juice	<i>Citrus limon</i>	Nimbu	Limbu
4.	Fresh tamarind leaves	<i>Tamarindus indica</i>	Imli	Chincha
5.	Whole harda powder	<i>Terminalia chebula</i>	Harad	Hirda

Table.2 Percentage of absorption for extraction time of annatto dye on cotton fabric

Sr. No.	Dye extraction time	OD before dyeing	OD after dyeing	% of absorption
1.	15 mins	0.499	0.477	4.4% **
2.	30 mins	0.595	0.577	3.0%
3.	45 mins	0.594	0.574	3.3%
4.	60 mins	0.641	0.619	3.4%
5.	75 mins	0.607	0.595	1.9% *
6.	90 mins	0.730	0.706	3.2%

(*lowest % absorption, ** highest % absorption)

Table.3 Percentage of absorption of dye amount for annatto seeds on cotton fabric

Sr. No.	Dye amount	OD before dyeing	OD after dyeing	% of absorption
1.	10 gm	0.629	0.622	1.11%
2.	15 gm	0.601	0.566	5.82% **
3.	20 gm	0.669	0.659	1.49%
4.	25 gm	0.777	0.772	0.38% *
5.	30gm	0.925	0.915	1.08%

(*lowest % absorption, ** highest % absorption)

Table.4 Percentage of absorption in various dyeing time for annatto seeds on cotton fabric

Sr. No.	Dyeing time	OD before dyeing	OD after dyeing	% of absorption
1.	15 mins	0.537	0.533	0.74%
2.	30 mins	0.550	0.537	2.36%
3.	45 mins	0.569	0.550	3.33% **
4.	60 mins	0.727	0.729	0.41%
5.	75 mins	0.563	0.555	1.42%
6.	90 mins	0.769	0.767	0.26% *

(*lowest % absorption, ** highest % absorption)

Table.5 % absorption of various amount of Alum with annatto seeds on Cotton

Sr. No.	Mordant amount	OD before dyeing	OD after dyeing	% of absorption
1.	2 gm	0.508	0.368	27.55%
2.	4 gm	0.508	0.281	44.68% **
3.	6 gm	0.508	0.344	32.28%
4.	8 gm	0.508	0.359	29.33%
5.	10 gm	0.508	0.367	27.75%
6.	12 gm	0.508	0.403	20.66% *

(*lowest % absorption, ** highest % absorption)

Table.6 % absorption of various amount of Aloe Vera gel with annatto seeds on Cotton

Sr. No.	Mordant amount	OD before dyeing	OD after dyeing	% of absorption
1.	2 gm	0.630	0.510	19.04%
2.	4 gm	0.630	0.539	14.44%
3.	6 gm	0.630	0.563	10.63%
4.	8 gm	0.630	0.559	11.26%
5.	10 gm	0.630	0.589	6.50% *
6.	12 gm	0.630	0.498	20.95% **

(*lowest % absorption, ** highest % absorption)

Table.7 % absorption of various amount of Lemon Juice with annatto seeds on Cotton

Sr. No.	Mordant amount	OD before dyeing	OD after dyeing	% of absorption
1.	2 gm	0.787	0.634	19.44%
2.	4 gm	0.787	0.630	19.94% **
3.	6 gm	0.787	0.716	9.02%
4.	8 gm	0.787	0.690	12.32%
5.	10 gm	0.787	0.701	10.92%
6.	12 gm	0.787	0.759	3.55% *

(*lowest % absorption, ** highest % absorption)

Table.8 % absorption of various amount of Tamarind leaves with annatto seeds on Cotton

Sr. No.	Mordant amount	OD before dyeing	OD after dyeing	% of absorption
1.	2 gm	0.719	0.621	13.63% **
2.	4 gm	0.719	0.684	4.86% *
3.	6 gm	0.719	0.679	5.56%
4.	8 gm	0.719	0.660	8.20%
5.	10 gm	0.719	0.673	6.39%
6.	12 gm	0.719	0.680	5.42%

(*lowest % absorption, ** highest % absorption)

Table.9 % absorption of various amount of Harda with annatto seeds on Cotton

Sr. No.	Mordant amount	OD before dyeing	OD after dyeing	% of absorption
1.	2 gm	0.857	0.801	6.53% **
2.	4 gm	0.857	0.807	5.83%
3.	6 gm	0.857	0.831	3.03%
4.	8 gm	0.857	0.810	5.48%
5.	10 gm	0.857	0.842	1.75%
6.	12 gm	0.857	0.845	1.40% *

(*lowest % absorption, ** highest % absorption)

Table.10 % Dye absorption of Pre mordanting method for Annatto Seeds on Cotton

Sr. No.	Mordant amount	Optimized amount of mordant used	OD before dyeing	OD after dyeing	% of absorption
1.	Alum	4 gm	0.874	0.797	8.81% *
2.	Aloe Vera	12 gm	0.874	0.455	47.94%
3.	Lemon Juice	4 gm	0.874	0.431	50.68%
4.	Tamarind leaves	2 gm	0.874	0.487	50.57%
5.	Harda	2 gm	0.874	0.432	50.74% **

(*lowest % absorption, ** highest % absorption)

Plate.1 Annatto seeds, selected natural dye



Plate.2 Alum crystals	Plate.3 Leaves of Aloe Vera	Plate.4 Harda powder
		

Plate.5 Fresh Lamon	Plate.6 Tamarind leaves
	

Plate.7 Samples of optimization of Dye extraction time of Annatto Dye on Cotton

Process	Sample	Pantone No.
Optimisation of dye extraction time		
Extraction of Annatto seeds dye for 15 min *		PANTONE 1565 C
Extraction of Annatto seeds dye for 30 min		PANTONE 1485 C
Extraction of Annatto seeds dye for 45 min		PANTONE 1485 C
Extraction of Annatto seeds dye for 60 min		PANTONE 1565 C
Extraction of Annatto seeds dye for 75 min		PANTONE 1565 C
Extraction of Annatto seeds dye for 90 min		PANTONE 1485 C

Plate.8 Samples of optimization of Dye Amount of Annatto Dye on Cotton

Process	Sample	Pantone No.
Optimisation of dye amount		
Extraction of 10 gm Annatto seeds dye for 15 min		PANTONE 2011 C
Extraction of 15 gm Annatto seeds dye for 15 min *		PANTONE 2017 C
Extraction of 20 gm Annatto seeds dye for 15 min		PANTONE 2017 C
Extraction of 25 gm Annatto seeds dye for 15 min		PANTONE 713 C
Extraction of 30 gm Annatto seeds dye for 15 min		PANTONE 2017 C

Plate.9 Samples of optimization of Dyeing time of Annatto Dye on Cotton

Optimisation of dyeing time		
Dyeing of Annatto seeds dye for 15 min		PANTONE 163 C
Dyeing of Annatto seeds dye for 30 min		PANTONE 163 C
Dyeing of Annatto seeds dye for 45 min		PANTONE 163 C
Dyeing of Annatto seeds dye for 60 min		PANTONE 2011 C
Dyeing of Annatto seeds dye for 75 min		PANTONE 2011 C
Dyeing of Annatto seeds dye for 90 min		PANTONE 2011 C

Plate.10 Samples of optimization of Alum Mordant Amount for Annatto Dye on Cotton

Optimisation of mordant amount		
Mordanting of Annatto dyed fabric with 2 gm Alum (Simultaneous mordanting)		PANTONE 155 C
Mordanting of Annatto dyed fabric with 4 gm Alum * (Simultaneous mordanting)		PANTONE 155 C
Mordanting of Annatto dyed fabric with 6 gm Alum (Simultaneous mordanting)		PANTONE 2015 C
Mordanting of Annatto dyed fabric with 8 gm Alum (Simultaneous mordanting)		PANTONE 155 C
Mordanting of Annatto dyed fabric with 10 gm Alum (Simultaneous mordanting)		PANTONE 2015 C
Mordanting of Annatto dyed fabric with 12 gm Alum (Simultaneous mordanting)		PANTONE 155 C

Plate.11 Samples of optimization of Aloe Vera gel Mordant Amount for Annatto Dye on Cotton

Mordanting of Annatto dyed fabric with 2 gm Aloe Vera gel (Simultaneous mordanting)		PANTONE 1485 C
Mordanting of Annatto dyed fabric with 4 gm Aloe Vera gel (Simultaneous mordanting)		PANTONE 1485 C
Mordanting of Annatto dyed fabric with 6 gm Aloe Vera gel (Simultaneous mordanting)		PANTONE 156 C
Mordanting of Annatto dyed fabric with 8 gm Aloe Vera gel (Simultaneous mordanting)		PANTONE 156 C
Mordanting of Annatto dyed fabric with 10 gm Aloe Vera gel (Simultaneous mordanting)		PANTONE 156 C
Mordanting of Annatto dyed fabric with 12 gm Aloe Vera gel * (Simultaneous mordanting)		PANTONE 156 C

Plate.12 Samples of optimization of Lemon Juice Mordant Amount for Annatto Dye on Cotton

Mordanting of Annatto dyed fabric with 2 gm Lemon juice (Simultaneous mordanting)		PANTONE 155 C
Mordanting of Annatto dyed fabric with 4 gm Lemon juice * (Simultaneous mordanting)		PANTONE 2015 C
Mordanting of Annatto dyed fabric with 6 gm Lemon juice (Simultaneous mordanting)		PANTONE 155 C
Mordanting of Annatto dyed fabric with 8 gm Lemon juice (Simultaneous mordanting)		PANTONE 155 C
Mordanting of Annatto dyed fabric with 10 gm Lemon juice (Simultaneous mordanting)		PANTONE 155 C
Mordanting of Annatto dyed fabric with 12 gm Lemon juice (Simultaneous mordanting)		PANTONE 155 C

Plate.13 Samples of optimization of tamarind Mordant Amount for Annatto Dye on Cotton

Mordanting of Annatto dyed fabric with 6 gm Tamarind leaves (Simultaneous mordanting)		PANTONE 157 C
Mordanting of Annatto dyed fabric with 8 gm Tamarind leaves (Simultaneous mordanting)		PANTONE 157 C
Mordanting of Annatto dyed fabric with 10 gm Tamarind leaves (Simultaneous mordanting)		PANTONE 156 C
Mordanting of Annatto dyed fabric with 12 gm Tamarind leaves (Simultaneous mordanting)		PANTONE 156 C

Plate.14 Samples of optimization of Harda powder Mordant Amount for Annatto Dye on Cotton

Mordanting of Annatto dyed fabric with 2 gm Harda powder * (Simultaneous mordanting)		PANTONE 1365 C
Mordanting of Annatto dyed fabric with 4 gm Harda powder (Simultaneous mordanting)		PANTONE 1355 C
Mordanting of Annatto dyed fabric with 6 gm Harda powder (Simultaneous mordanting)		PANTONE 1355 C
Mordanting of Annatto dyed fabric with 8 gm Harda powder (Simultaneous mordanting)		PANTONE 1355 C
Mordanting of Annatto dyed fabric with 10 gm Harda powder (Simultaneous mordanting)		PANTONE 1365 C
Mordanting of Annatto dyed fabric with 12 gm Harda powder (Simultaneous mordanting)		PANTONE 1355 C

The study further revealed the importance of selecting appropriate mordants. Among the five natural mordants tested (Alum, Aloe Vera, Lemon juice, Tamarind leaves, and Harda powder), Tamarind leaves and Harda powder demonstrated the highest dye absorption in pre-mordanting treatments, suggesting their potential for enhancing the dyeing process. Additionally, post-mordanting was found to cause slight color shifts, emphasizing the need for careful mordant application to maintain color integrity. Overall, the results highlight the feasibility of utilizing natural dyes like Annatto for sustainable cotton dyeing, with optimized processes that

can reduce environmental impact and resource consumption. This research contributes valuable insights for the growing field of sustainable textile production and supports the shift towards greener alternatives in the fashion and textile industries.

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Author Contributions

Dr. Anshu Sharma: Conceived the original idea and designed the model and wrote the manuscript.

Data Availability

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethical Approval Not applicable.

Consent to Participate Not applicable.

Consent to Publish Not applicable.

Conflict of Interest The authors declare no competing interests.

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