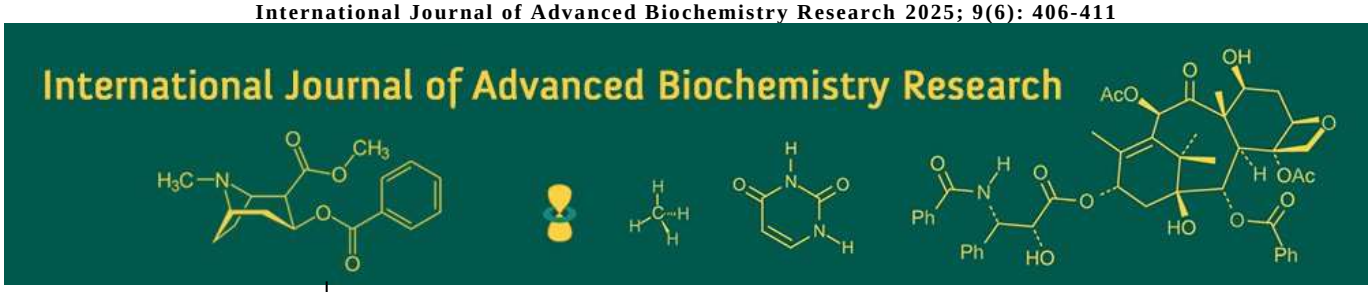

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Formulation and evaluation of herbal toothpaste incorporating *Zanthoxylum armatum*

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Abstract
Increased need for safe and natural substitutes for synthetic oral care products has generated interest in the development of herbal preparations. In this research, the development and testing of a toothpaste formula based on *Zanthoxylum armatum*, known as Timur or Toothache tree, were examined. The extracts of the bark and fruit of *Z. armatum* were blended with a toothpaste base and analyzed for different physicochemical parameters such as pH, foaming capacity, abrasivity, spread, and stability. Microbiological evaluation was carried out against regular oral pathogens like Streptococcus mutans and Lactobacillus acidophilus by the agar well diffusion test. The formula showed reasonable organoleptic characteristics and showed high antimicrobial activity similar to that of commercial toothpaste. The results validate the promise of *Z. armatum* as an active ingredient in herbal toothpaste products, providing a natural and effective method for oral care with lower risk of adverse effects compared to synthetic chemicals.

Keywords: *Zanthoxylum armatum*, herbal toothpaste, antimicrobial activity, oral hygiene, natural formulation

Introduction
Oral hygiene is an important aspect of overall health, with inadequate oral care being a causative factor for many dental conditions such as dental caries, gingivitis, periodontitis, and halitosis. Oral hygiene has been maintained traditionally using mechanical and chemical approaches, and toothpaste is the most utilized oral care product used worldwide [1]. Conventional toothpaste systems always consist of artificial antimicrobial chemicals, abrasives, surfactants, and fluoride compounds. Although these synthetic agents are potent agents in ensuring oral health, long-term usage has been linked with side effects of mucosal irritation, staining of the teeth, changing of the perception of taste, and in certain situations, systemic toxicity [2].

As a response to such issues, there is a rising trend towards herbal and natural remedies for dental care. Herbal toothpaste composition takes advantage of the medicinal properties of herbaceous plants, providing an environmentally friendly, biocompatible, and safer method of oral hygiene [3]. Among the many medicinal plants that have been studied for oral use, *Zanthoxylum armatum* DC. (Rutaceae), known as Timur, is of special interest because of its vast range of pharmacological activities [4].

Zanthoxylum armatum is a deciduous, fragrant shrub or small tree that is native to the subtropical and temperate parts of the Himalayas, especially in India, Nepal, Bhutan, and China. The plant has been used traditionally in Ayurvedic and Unani systems of medicine to treat toothache, gastrointestinal conditions, respiratory problems, and microbial infection [6]. Fruits, bark, and seeds of the plant contain high amounts of bioactive compounds such as alkaloids, flavonoids, essential oils, and lignans. These constituents impose a variety of valuable properties like antimicrobial, anti-inflammatory, analgesic, astringent, and antioxidant activities [10].

Various studies have reported the antimicrobial activity of *Z. armatum* against oral bacteria. For instance [4], exhibited substantial inhibitory activity of *Z. armatum* fruit extract against Streptococcus mutans, Lactobacillus acidophilus, and *Candida albicans*, indicating its possible involvement in the prevention of dental caries and oral candidiasis.

In a similar vein, [13] documented the linalool, limonene, and xanthoxylin presence in *Z. armatum's* essential oil, all of which have strong antimicrobial and anti-inflammatory properties applicable to dental care.

Additionally, the astringent and analgesic properties of the plant are especially beneficial in controlling bleeding gums and toothache. Its use as a chewing stick or even as a powder for cleaning the teeth further serves to indicate its cultural and therapeutic significance in oral care upkeep [14].

Despite these promising properties, the use of *Z. armatum* in contemporary oral care products is underexplored. Not many commercial products take advantage of its bioactive capacity, and there are few standardized formulations and thorough scientific assessment in this regard [15].

This study not only aims to fill the gap between ancient knowledge and contemporary pharmaceutical technology but also complements the growing consumer demand for environmentally friendly and health-oriented products. Through the systematic assessment of the efficacy and safety of toothpaste based on *Z. armatum*, this work contributes to the creation of new, plant-based oral care products that are both effective and sustainable [16].

Background of the Study

Oral health is important to overall health, and the prevention of certain dental problems, including plaque development, gingivitis, and halitosis, depends on adequate oral hygiene. Traditional toothpastes contain included many synthetic ingredients that may have a variety of side effects, such as irritation of the mucosal membranes or altered taste. Due to these concerns, there has been growing popularity in the herbal movement that stresses safety as well as efficiency. *Zanthoxylum armatum* DC (Timur or Toothache Tree) is a medicinal plant with a traditional history of use in Ayurvedic and folk medicines for the antimicrobial, anti-inflammatory, and analgesic properties of different parts of the plant. The fruits and bark of *Zanthoxylum armatum*, in particular, have been documented for treatment of toothaches and infections. Despite its use in ethnomedicine, there have been few studies that investigate it for use in nontraditional, modern dental products. This study will formulate and evaluate an herbal toothpaste which incorporates *Zanthoxylum armatum*, to investigate the potential for this product to act as a natural, safer, and effective means for enhancing oral hygiene, as well as preventing oral diseases.

About *Zanthoxylum armatum*

Scientific Name: *Zanthoxylum armatum*

Fig 1: Fruits of *Zanthoxylum armatum*

Fig 2: Leaves of *Zanthoxylum armatum*

Fig 3: Dried Seed of Zanthoxylum

Fig 4: Dried Seed of Zanthoxylum

Table 1: Taxonomic Classification:

S. No	Rank	Classification
1.	Kingdom	Plantae
2.	Subkingdom	Tracheobionta (Vascular plants)
3.	Division	Magnoliophyta (Angiosperms)
4.	Class	Magnoliopsida (Dicotyledons)
5.	Order	Sapindales
6.	Family	Rutaceae
7.	Genus	Zanthoxylum
8.	Species	*Zanthoxylum armatum* DC.

Botanical Description

- **Habit:** A spiny, aromatic shrub or small deciduous tree, growing up to 6-7 meters in height.
- **Stem:** Woody, straight or slightly bent, armed with conical spines.
- **Leaves:** Alternate, compound (typically with 3-9 leaflets), odd-pinnate; leaflets are ovate to lanceolate, aromatic when crushed, and have a leathery texture.
- **Flowers:** Unisexual, small, greenish-white to yellowish, borne in axillary or terminal panicles; dioecious plant (male and female flowers on separate plants).
- **Fruits:** Small follicles, subglobose to ellipsoid, reddish to black when mature, splitting open to release shiny black seeds.
- **Seeds:** Hard, round, and black with a pungent taste.

Geographical Distribution

- **Native to:** The Himalayas and other mountainous regions of South and Southeast Asia.
- **Countries:** India (particularly in Himachal Pradesh, Uttarakhand, Sikkim, and North-East), Nepal, Bhutan, China (Yunnan, Sichuan), Pakistan, and Northern Myanmar.
- **Altitude range:** Commonly grows at elevations between 900-2,000 meters above sea level.
- **Habitat:** Prefers well-drained, rocky, or sandy soils in subtropical and temperate forest regions.

Ethnobotanical and Traditional Uses: *Zanthoxylum armatum* has been used in several traditional medicine systems:

- **Ayurveda:** Known as Tumburu; used for dental ailments, as a digestive stimulant, carminative, and for treating respiratory issues like cough and asthma.
- **Traditional Chinese Medicine (TCM):** The dried fruit is used as a warming agent, pain reliever, and digestive tonic.
- **Folk Medicine:** Twigs used as natural toothbrushes; fruits chewed to relieve toothache and freshen breath.

Phytochemical Constituents

- **Alkaloids:** Berberine, skimmianine
- **Flavonoids:** Quercetin, kaempferol
- **Lignans:** Sesamin, asarinin
- **Essential oils:** Linalool, limonene, cineole, estragole, xanthoxylin
- **Tannins:** Gallic acid derivatives
- Saponins

Table 2: Pharmacological Activities:

S. No	Activity	Description	References
1.	Antimicrobial	Active against oral and gut pathogens such as Streptococcus mutans, *E. coli*, and *Candida albicans*.	4, 13
2.	Anti-inflammatory	Inhibits COX enzymes and reduces edema in animal models.	6
3.	Analgesic	Provides local numbing and pain relief, especially in toothache.	10
4.	Antioxidant	Scavenges free radicals; contains phenolic compounds.	12
5.	Anthelmintic	Effective against intestinal parasites.	24
6.	Insecticidal	Essential oil shows larvicidal activity.	13
7.	Digestive stimulant	Enhances salivation and gastrointestinal activity.	21

Parts Used

- **Fruits:** Mostly used in traditional and modern applications.
- **Seeds:** Occasionally used in spice mixtures.
- **Bark and twigs:** Used as toothbrushes or chewed directly for oral hygiene.
- **Leaves:** Less commonly used, but may contain aromatic compounds.

Materials and Methodology

Table 3: List of Materials

S. No	Ingredient	Function	Quantity per 100 g formulation
1.	*Zanthoxylum armatum* extract	Active antimicrobial agent	5 g
2.	Sorbitol	Humectant and sweetener	10 g
3.	Glycerin	Humectant and base	15 g
4.	Calcium carbonate	Mild abrasive	30 g
5.	Sodium lauryl sulfate (SLS)	Surfactant and foaming agent	1.5 g
6.	Carboxymethyl cellulose (CMC)	Thickener and binder	2 g
7.	Clove oil	Flavouring agent and analgesic	0.5 g
8.	Sodium benzoate	Preservative	0.2 g
9.	Distilled water	Solvent and vehicle	q.s. up to 100 g

Methodology

Drying and Powdering

- The collected fruits were washed, shade-dried for 7 days, and pulverized using an electric grinder.
- The coarse powder was sieved (mesh #40) and stored in an air-tight container.

Preparation of Extract: [Harborne *et al.*, 1998; Raaman, 2006]

Materials Required

- Dried powdered fruits of *Zanthoxylum armatum*-100 g
- Solvent-Ethanol (95%)-500 mL
- Whatman No. 1 filter paper
- Soxhlet apparatus (including thimble, round-bottom flask, condenser)
- Heating mantle or water bath
- Rotary evaporator
- Analytical balance

- Air-tight amber glass container

Procedure

Loading into Soxhlet Apparatus

- 100 g of powdered fruit was placed inside a cellulose thimble.
- The thimble was inserted into the main chamber of a Soxhlet extractor.

Solvent Selection and Extraction

- Ethanol (95%) was chosen for its polarity, safety, and ability to dissolve both polar and non-polar phytochemicals.
- 500 mL of ethanol was added to a 1,000 mL round-bottom flask connected to the Soxhlet extractor.

Heating and Recirculation

- The Soxhlet apparatus was heated at a temperature of 60-70°C using a heating mantle.
- Continuous extraction was carried out for 6-8 hours or until the solvent in the siphon tube appeared colourless, indicating exhaustive extraction.

Concentration of Extract

- The ethanol extract was concentrated using a rotary evaporator under reduced pressure at 40-50°C to remove excess solvent.
- The semi-solid crude extract was further dried in a desiccator over anhydrous silica gel.

Storage

- The dried extract was weighed, yield calculated, and stored in china dish at 4°C until further use.

Formulation of Herbal Toothpaste

Base Preparation

- In a clean mortar, calcium carbonate and CMC were mixed thoroughly with glycerine and sorbitol to form a homogeneous paste.

Incorporation of Extract

- The prepared *Z. armatum* extract (5 g per 100 g formulation) was mixed into the base using geometric dilution.

Addition of Additives

- Sodium lauryl sulfate was dissolved in a small volume of distilled water and added gradually.
- Peppermint oil and sodium benzoate were added last with gentle stirring.

Final Adjustment

- The total weight was adjusted to 100 g with distilled water to achieve desired consistency.
- The paste was homogenized and stored in air tight container.

Evaluation

Organoleptic Evaluation: Organoleptic properties were assessed using visual and sensory inspection by a panel of five evaluators. Parameters such as color, odor, taste, texture, and appearance were recorded [20].

Table 4: Organoleptic Evaluation

S. No	Parameter	Method Used
1.	Colour	Visual observation
2.	Odor	Sensory evaluation
3.	Taste	Taste panel
4.	Texture	Manual tactile inspection
5.	Appearance	Visual examination

Physicochemical Evaluation: The formulated toothpaste was evaluated for several physicochemical parameters, as summarized below

Table 5: Physicochemical Evaluation

Parameter	Methodology
pH (1% solution)	Digital pH meter
Foaming ability (%)	Cylinder shake method (after 1 min shaking)
Spreadability (g·cm/s)	Two-glass slide method
Viscosity (cPs)	Brookfield viscometer (Spindle No. 64, 10 rpm)
Moisture content (%)	Hot air oven at 105°C
Grittiness	Sensory test

Methodological Notes

- pH Measurement: 1 g of toothpaste was dispersed in 10 mL distilled water and measured using a calibrated digital pH meter.
- Foaming Ability: 1 g toothpaste was mixed in 50 mL distilled water, shaken in a graduated cylinder, and foam height measured.
- Spreadability: 1 g of sample placed between glass slides under 500 g weight, time to spread measured.
- Viscosity: Brookfield viscometer used at room temperature.

Results and Discussion

Table 5: Organoleptic Evaluation and Observations

Parameter	Observation	Method Used
Colour	Light brown with greenish tint	Visual observation
Odor	Characteristic spicy and aromatic fragrance	Sensory evaluation
Taste	Pungent with a mild tingling effect	Taste panel
Texture	Smooth, non-gritty	Manual tactile inspection
Appearance	Homogeneous, glossy paste	Visual examination

Table 6: Physicochemical Evaluation and Observations

Parameter	Methodology	Observed Value	Acceptable Range
pH (1% solution)	Digital pH meter	7.20±0.05	6.5-8.0
Foaming ability (%)	Cylinder shake method (after 1 min shaking)	45%	≥ 30%
Spreadability (g·cm/s)	Two-glass slide method	10.5 g·cm/s	8-12 g·cm/s
Viscosity (cPs)	Brookfield viscometer (Spindle No. 64, 10 rpm)	45,000±500 cPs	30,000-60,000 cPs

Moisture content (%)	Hot air oven at 105 °C	28.3%	≤ 30%

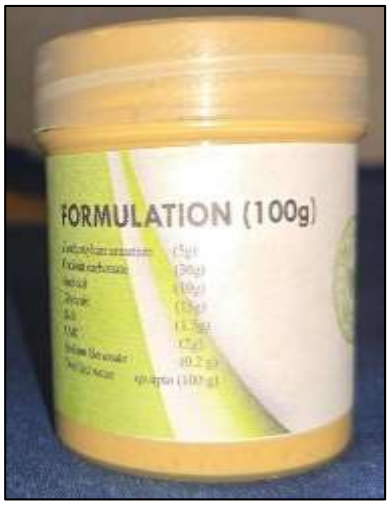

Fig 5: Herbal Toothpaste

Fig 6: Toothpaste Formulation

Discussion

The formulated herbal toothpaste exhibited favourable organoleptic and physicochemical characteristics, indicating its potential as a safe and effective dental hygiene product. The organoleptic evaluation showed that the toothpaste had an appealing light brown colour, characteristic aromatic odour, and a spicy taste typical of *Zanthoxylum armatum*, which may enhance user acceptability. The smooth texture and homogeneous appearance further suggest good formulation aesthetics and uniformity. In terms of physicochemical properties, the Ph of the formulation was found to be 7.2, which falls within the acceptable neutral to slightly basic range for oral products and is safe for prolonged use without causing enamel erosion or mucosal irritation. The foaming ability was 45%, indicating sufficient cleansing action, which is important for mechanical removal of debris and microbial biofilm. The spreadability was recorded at 10.5 g·cm/s, suggesting ease of application on the toothbrush without excess resistance. Viscosity of the formulation (45,000 cPs) ensured appropriate consistency, neither too thick nor too runny, maintaining user comfort and stable product integrity. Grittiness was absent, indicating proper milling and filtration of abrasive agents, which minimizes the risk of enamel abrasion.

Conclusion

The present study successfully formulated and evaluated an herbal toothpaste using the ethanolic extract of *Zanthoxylum armatum* DC, a medicinal plant known for its antimicrobial, anti-inflammatory, and analgesic properties. The formulation process incorporated natural and safe excipients to develop a toothpaste with favourable organoleptic properties and desirable physicochemical characteristics, including appropriate pH, viscosity, spreadability, and foaming ability. The organoleptic evaluation confirmed that the toothpaste had an acceptable taste, texture, colour, and aroma, which are important factors in user acceptability. The absence of grittiness, optimal moisture content, and favourable stability profile across different storage conditions further suggest the robustness and safety of the formulation.

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