STABILIZED CASSAVA GEL AND ITS PRODUCTION TECHNIQUE

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RP: NG/PT/NC/2021/5831
Date of Patent: 11/12/2021
Date of Sealing: 17/12/2021

President of the Federal Republic of Nigeria and Commander-in-chief of the Armed Forces
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Whereas a request for the grant of a patent has been made by: **CENTRE FOR INTELLECTUAL PROPERTY AND COMMERCIALIZATION (CIPPaC) , DR. AMOO, KEHINDE, DR. OMONIYI TEMIDAYO EMMANUEL, PROF. ONILUDE, MUSILIU ADE ALL OF DEPARTMENT OF WOOD PRODUCTS ENGINEERING, UNIVERSITY OF IBADAN *C/O* AWEFORTE ASSOCIATES, SUITE 24, FAITH PLAZA, LOKOGOMA, ABUJA .**

For the sole use and advantage of an invention for: **Stabilized Cassava-Based Gel and its Production Techniques**

AND WHEREAS the Federal Government being willing to encourage all invention which may be for public good, is pleased to accede to the request:

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04 February, 2022.

Dr. AMOO, Kehinde
Department of Wood Products Engineering,
University of Ibadan, Ibadan, Nigeria

Dear Sir,

RE: ISSUANCE OF PATENTS CERTIFICATE THROUGH (CIPPAC)

Title: Stabilized Cassava-Based Gel and Its Production Technique

We wish to congratulate you on the issuance of Patent Certificate (NG/PT/NC/2021/5831).

The Centre for Intellectual Property Protection and Commercialization reviewed the manuscripts and facilitated the certification in Patent registry office, Abuja. The issuance of the certificate is an indication of the innovative contents of your work, and we hope this can be commercialized through CIPPaC.

We wish to encourage you to keep up the good work.

Once again congratulations.

Yours sincerely,

04/02/2022

Dr. O. T. Okareh
Ag. Director of CIPPaC

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(a) STABILIZED CASSAVA-BASED GEL AND ITS PRODUCTION TECHNIQUE

(b) State (in full) name, address and nationality of applicant or applicants as in application form.

(b) I/We 1.Dr. AMOO, Kehinde (2) Dr. OMONIYI, Temidayo Emanuel (3) Prof. ONILUDE, Musiliu Ade
Department of Wood Products Engineering, , University of Ibadan, Ibadan, Nigeria

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do hereby declare the invention, for which I/we pray that a patent may be granted to me/us, and the method by which it is to be performed, to be particularly described in and by the following statement

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APPLICATION FOR PATENT

STABILIZED CASSAVA GEL AND ITS PRODUCTION TECHNIQUE

BY

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Abstract

An invention in the production of a stabilised, and durable gel (having a long shelf-life of about two years), from cassava starch for applications as sizing-agent in pulp and paper making, wood-composites production, textile/laundry services and other relevant uses is presented. The new product called KAMOGEL is made by combining the pre-mixed and pre-determined quantities of cassava starch, water, preservatives, stabiliser/emulsifier, and essential oil sequentially.

Background to the Invention

A typical sizing-agent formulation usually consists of a substantial amount of adhesive components, which may include natural starch, as the active sizing ingredient, and other additives which may include emulsifier, preservatives, stabiliser, polyvinyl compound, essential oil and borax. These are engaged to initiate a gluing bond with substrate's surface. This invention demonstrates the sizing-agent formulation that is applicable in the pulp and paper industry and other related fields. Typical sizing paste configurations are principally based on a natural polymer, usually starch, but may also include partially or wholly appropriate artificial adhesive polymers, such as polyvinyl and others. This creation is mainly a *size* that is non-hazardous, and may be used in contact with human body-parts

According to Gunorubon (2012), adhesives are materials that facilitate adherence or sticking of two surfaces without distortion or failure. This process is termed adhesion (Baumann and Conner, 2003). Adhesives are either in natural or synthetic form. The natural ones are glues from animal, casein, natural gums/resins, sodium silicates and vegetable. Most

importantly, the vegetable adhesives are starch-based and are generally produced from starches and its derivatives (Kennedy, 1989). Starch applications as ingredient in adhesive-making is more advantageous than other materials in terms of its diversity, versatility and accessibility (FAO, 1983; Agboola,*et. al*., 1990).

Starch is mainly produced from crops which include sweet potatoes, maize, wheat, rice, yam or cassava. Among the afore-mentioned, cassava is the most advantageous for starch making due to its acceptable characteristics such as high clarity level, exceptional thickening features, a neutral taste, appropriate textural appearances, relative economic advantage and extraordinary starch concentration (Masamba *et. al*., 2001; Tonukari, 2004).

However, the efficiency of a sizing product is measured by the bonding capacity, workability, rational setting characteristics, moisture resistance, anti-aging properties, and durability (Finn, 1990). The sizing adhesive created from cassava starch tends to possess desirable texture, mildness, viscosity, relative stability and neutrality (Masamba, *et. al*., 2003). The key downside noticed by using starch as an sizing and adhesive ingredient is the stability, and resistance (FAO, 1983). This present invention therefore aims at solving the above described problems and provides improvements in order to increase the adhesive product's shelf life, performance and durability.

1.0 Brief Description of the Invention

The objective of this invention was to provide a paste (sizing adhesive), with a long shelf-life (about two years), from raw cassava starch as a better alternative to the existing cassava gel which has a short shelf-life (about five days); made of cassava starch and other additives used in the pulp and paper, wood composites and textile industry. The invented gel is an eco-friendly and suitable invention in the starching of cotton and combination-fibre fabrics. The product can be applied directly or in diluted form without compromising its effectiveness. It is a finished product that saves time, energy, money and is easy to use. There is no need for boiling of water for its application. The product is adequately viscous, smooth to mix as it does not form lumps during mixing. Also, it is a stable and durable product that does not stagnate, separate or change colour during storage.

2.0 Description of the state of the art

The yearning for more durable, stable, resistant, eco-friendly and alternative sizing products to be used in pulp and paper, wood composite, textile, environmental and other engineering industries motivated this invention. Cassava is available crop in large quantities. Starch, especially from cassava, has been used to enhance stiffness in paper and textile industries for decades. However, the extraction of starch from cassava, especially the preparation and application of the starch (gel), comes with some challenges. Cassava gel preparation takes time, energy and resources. Also, the gel produced is easily susceptible to attack by micro-organism and get deteriorated. As a result, the gel loses its adhesive properties and becomes watery, non-sticky, infected and malodorous after few days. Therefore, there is a need to stabilise and preserve the gel from biological attack to increase its shelf-life for frequent and future use. Consequently, cassava starch, as the primary ingredient in this invention (product), guarantees consistent supplies of raw material, encourages patronage of local contents, reduces capital flight, and creates employment and boosts the country's economy.

Starch exists naturally in various agricultural products as the principal food constituent and a regular carbohydrate polymer broadly employed in diverse industrial applications. The papermaking and textile/laundry industries are presently being challenged to cut their production costs due to market competitions. Thus, applications of a cost-effective and high-efficient plant-based starch sizing product will profit the industries (Chen et al. 1998). Nevertheless, cassava gel exhibits high tack tendency, moderate penetration into the substrates, non-staining, non-bleeding and non-curling behaviour. It is believed to be smooth, and non-blocking under tropical circumstances. Also, cassava-based sizing products are employed due to their affordable cost and relative accessibility. Pastes, like that from cassava starch, for sizing, binding and sealing possess a steady viscosity, adequate bondability, and ability to set up fast. (Vishnuvarthanan and Rajeswari, 2013). According to Radley (1976), cassava starch-derived adhesive having lower solids concentration will set at a 24% rate faster than maize or any other starch base due to its tackiness and higher concentration of amylopectin.

Although many industries are employing maize and some other plant-based starch, it would, however, be appropriate to switch to the use of cassava starch due to many reasons

ranging from its high starch yield, the high tack capacity, accessibility and ease of starch extraction (Agboola et al. 1990; Richard et al. 1991; da Silva and Adam, 2006). Comparatively, extracting starch from cassava tubers is easier than maize and any other plant sources. The gel made from there is found to be tackier, more convenient to use, more viscous and exhibit better tensile strength than from any other plant origins (Radley, 1976). According to Jarowenko (1977), cassava starch has a lower setting time and a gelatinisation temperature of 49-70°C compared to maize (62-73°C). This trend shows that less heat will be needed to make a cassava-based gel, thus saving time, energy and cost.

Being from a polymeric compound, cassava gel as sizing additive provides an excellent affinity to polar substrate which includes cellulose and its derivatives (Jarowenko, 1977). Also, the process of cooking ruptures the cassava starch molecules to form a gel and reduces their tendencies to swell during and after pasting. Uncooked starch molecules, however, are firmly fused together and therefore cannot initiate an adhesion. Due to cooking of the cassava starch in water, its particles experience dispersal after their rupture. During the process, amylose and amylopectin particles are released and thereafter glue to the surface of the substrates to form a firm bond

At the early heating stage, an increase in viscosity of the gel is attributed to the starch particles starting to expand and amylose leaking out from the pellets. The highest viscosity occurs when the majority of the particles are completely inflated. At the elevated temperature, a fall in viscosity occurs due to the pellets breaking down. Continuous stirring not only solubilizes but also clips the amylopectin particles, which further causes a significant fall in molecular weight of amylopectin and motivates a succeeding viscosity fall. The cooling stage provides a second upsurge in gel viscosity as the amylose and amylopectin molecules begin to re-align (Lillford, 1997; Kearsley 1989; Thomas and Atwell 1999). In this regard, the stabilizer is found to intensify the water-holding capability of the gel and give it an increased molecular weight (Jin *et. al*., 2010).

Afterward, a reaction (retrogradation) takes place when the molecules of amylose and amylopectin are cooked; in the process the gelatinized starch then realign themselves as the gel cools. When natural starch is being cooked in water, the crystalline configuration of amylose and amylopectin molecules is nowhere to be found but they later absorb water to form a viscous solution. When the sticky solution is cooled for some period,

the direct fragments of amylose, and direct fragments of amylopectin particles retrograde and reposition themselves again to a more crystal-like assembly. The rectilinear chains position themselves parallel and as a result form hydrogen bonds. However, additives such as emulsifier and stabilizer as showcased in this present invention can decrease the retrogradation process, encourages film to dry quickly and inspires faster tackiness and adhesion in starch product.

3.0 Detailed Description of the Invention

The ingredients such as; cassava starch recovered from the effluent from a cassava processing establishment (preferably, TME 419 variety at 98% moisture content); the mineral filler (SINAPOL W115); the emulsifier/stabiliser (preferably, 250 HHBR, 7% moisture content, 0.6 g/ml Bulk Density); the essential oil (0.00083 w/w); and preservatives (Sodium chloride at 0.01 w/w and formaldehyde 27% v/v at 0.0083 w/w dosage); were obtained from the market and prepared according to Amoo et al. 2019. Before cassava gel formation, the mineral filler of a known mass was weighed into a container, and tap water of about 200% by weight of the filler was added and continuously mixed until all lumps dissolved and even solution formed. The solution was milky-white and moderately viscous. Also, the emulsifier of about 5% (w/w) of water was added slowly into cool and agitating tap water at room temperature. The mixture was then stirred continuously in a clockwise direction for 28 min (hydration time) until all swollen particles dissolved and even solution formed.

Subsequently, neat cassava starch of a known mass was mixed with ordinary tap water of equal mass to form homogeneous slurry at room temperature. Then, the water of 400% volume of the slurry was made to boil at 100 °c and allowed to simmer. The prepared slurry was gently poured into the boiled water of about 85 °c and stirred in a clockwise direction until a gel of even texture formed. The gel was allowed to cool to about 50°c, and the pre-mixed additives of pre-determined quantity were added to the cassava gel and mixed to homogeneity to form the KAMOGEL. Then, while still warm, the KAMOGEL was poured into an air-tight container and kept in a cool and dry place for future use. The production flow-chart of KAMOGEL is as shown in Fig.1

The KAMOGEL (Plate 1) quantity of about10% by weight of water is the least dosage recommended for adequate bonding and enhancement of the substrates' sorption, physical and mechanical properties, as may be used in pulp and paper, textile and other engineering

applications. The average setting time of the gel is 240±5 seconds. Dosage increase of the gel shows significant effect on bonding properties of the gel and significant enhancement of the substrates' physico-mechanical properties.

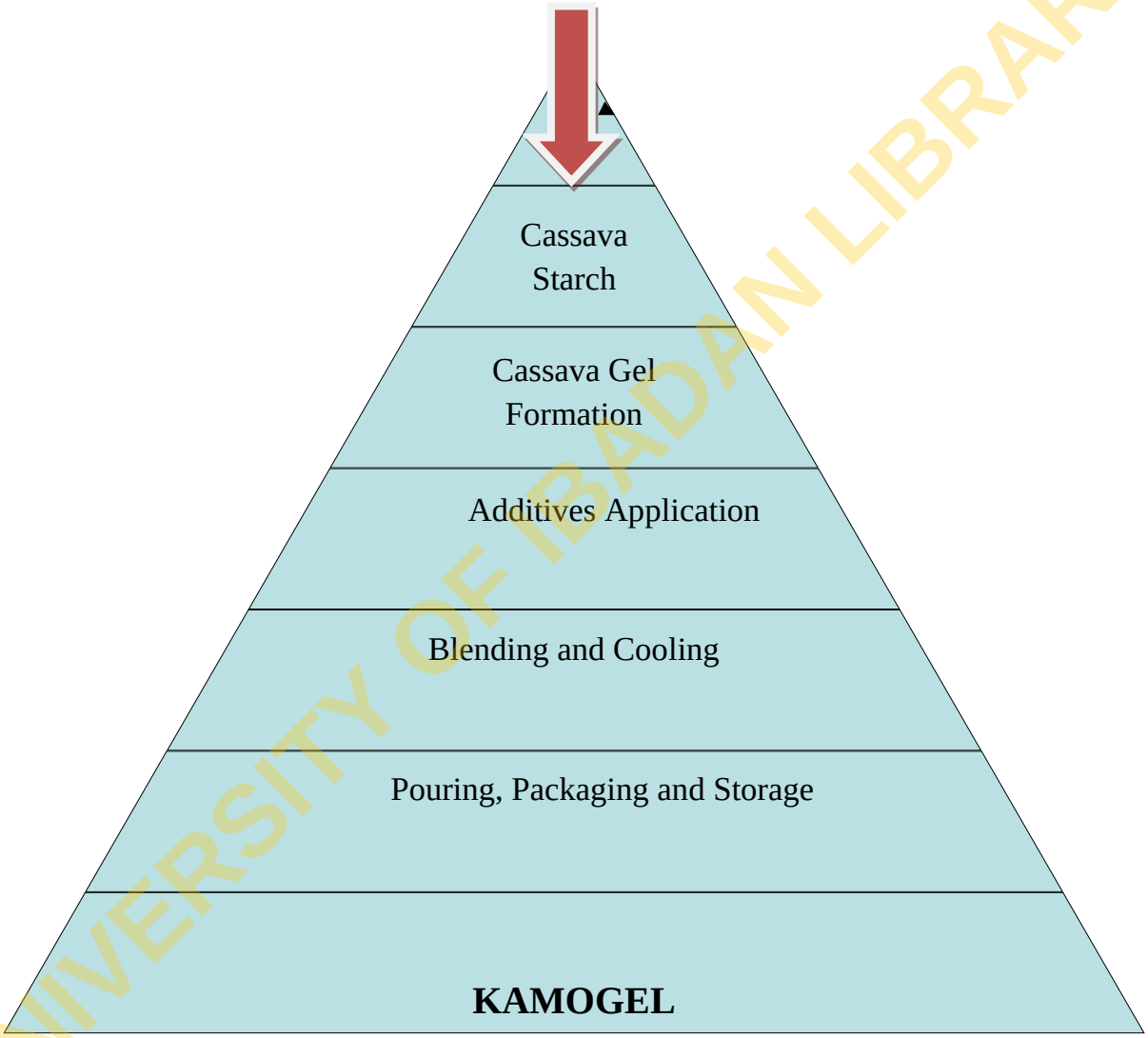

Figure 1: The Production Flow Chart of KAMOGEL

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Plate 1: Stabilised Cassava Gel (KAMOGEL)

4.0 Advantages of KAMOGEL over other Existing Gel.

The following are some of the advantages of the invented stabilised cassava gel and the existing un-stabilised cassava gel.

i. **Viscosity**: The invented product has a good percentage of solid contents that make them thick and viscous. Therefore, more volume of water may be used for their dilution without affecting the desired result.

ii. **Tackiness:** The product has a better performance at sticking, gumming and glueing to the surface of the substrates without draining off, hence increasing its stiffness.

iii. **Even Dissolution:** The product contains ingredients that make them mix evenly in water without forming lumps.

iv. **Long shelf life:** The addition of an adequate volume of preservatives to the product makes it resistant to bio-attack and therefore increases its shelf life (about two years).

v. **Stability:** The product does not stagnate, separate or change colour during storage. This quality is achieved with the addition of some specific ingredients to the formulation.

vi. **Cost:** The product is comparatively cheaper to acquire than the equal volume of other paste/adhesive in the market

vii. **Mildness:** The products contain natural starch as the primary ingredient. So, they are mild to use.

viii. **Convenience:** The product is a finished adhesive material that is ready for use. Hence, it saves time and energy.

ix. **High Dry-strength:** The addition of mineral filler inhibits the adhesive's bleeding tendencies and improves its dry strength over the existing ones.

x. **Good Smell:** The product is well-scented due to the inclusion of natural scent in the formulation

5.0 New KAMOGEL for Improved Industrial Applications

The following are some of the benefits of the invented product;

i. The product is easy to use, saves time, energy and money. No boiling of water needed for its application.

ii. It is made from a natural starch source, smooth to use (no lumps formed), and it is adequately viscous.

iii. It is an affordable product due to the use of cassava starch as the main ingredient.

iv. It is stable and durable and can stay for years without losing its adhesive properties. Also, it does not stagnate, separate and change colour during storage.

v. It is well perfumed to soothe the users.

vi. It has long shelf life (about two years) and resistance to bio-attack

The outstanding attributes of this product speak volume. The weaknesses of some other existing adhesive brands are corrected, and their perceived strengths improved upon in the invention.

6.0 Claims

My claims are:

I. The techniques for cassava starch recovery from effluent, production of gel from neat cassava starch, and eventual production of KAMOGEL from cassava gel having added the required additives as highlighted in this present invention.

II. The KAMOGEL production method which involves making a gel from raw cassava starch, mixing with pre-determined quantities of additives and stored in a cool and dry environment

III. The invention of an eco-friendly, durable, non-staining and stabilised cassava gel that has a long shelf life of at least two years without stagnation, separation, foul odour, and change of colour during storage. The product is called KAMOGEL and can be used as a cold-setting plant-based sizing additive in pulp and paper making, wood composite production, textile manufacture, environmental and other engineering applications.

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and

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6.0 References

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