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# Research Article

# CHEMISTRY OF WHEAT POLYSACCHARIDE BEAD; FORMULATION AND CHARACTERIZATION FOR IMPROVED QUALITY BEAD

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**ABSTRACT**

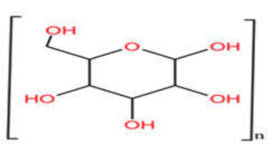
Beads are often small and spherical objects, made of clay, glass, paper, plastic, wood, or other suitable materials. They come in different colours, and usually perforated for a string to connect one with another; and they are usually used in making pieces of jewellery especially for beautification. This novel study was aimed at formulating and characterizing the production of improved quality bead from wheat flour. Hence, the chemistry of wheat flour polysaccharide bead involving formulation and characterization was carried out. Various composite materials such as zinc oxide (ZnO), titanium dioxide ($TiO_2$), calcium carbonate ($CaCO_3$), barium sulphate ($BaSO_3$), $CaCO_3$ + Plaster of Paris (POP) cement were used together with the base material differently. Results from the study showed that of all the composite materials tested, $TiO_2$ based bead gave a superior quality over its counterparts and over the conventional wheat flour bead, as weight increased > 9%, fragility (improved from 20min/300m$^{-s}$ – 40min/500m$^{-s}$), fastness property (improved from one month to over four months of testing period), reduced water absorption (improved from 4.02 – 0.78%), heat tolerance (improved from 50$^{o}$C –> 125$^{o}$C) and surface smoothness (improved from rough and unsymmetrical to smooth and symmetrical) and thus achieved a desirable product. The use of Fourier infra-red confirmed the addition of the $TiO_2$ to the wheat flour with a resultant superior quality of the bead.

***Keywords:*** bead, composite, materials, flour, optimization, polysaccharide, wheat, infra-red.

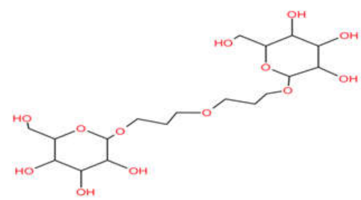
## INTRODUCTION

Bead is one of the earliest known forms of artistic expression, used in human body adornment and, an example is the shell beads of Blombos caves in South Africa (Jacobs *et al*., 2006). Generally, beads are made from both organic and inorganic materials. While the natural organics are known to include bones, corals, horns, ivory, seeds (e.g. tagua nuts), animal shells, and woods, the natural inorganics are known to include various types of stones, ranging from gemstones to common minerals, and metals. The oldest surviving synthetic materials used for bead making have generally been ceramics: pottery and glass. Although beads were also made from ancient alloys such as bronze and brass, yet the problem of oxidation, as expected, has confined them to archaeological spots. Beads types can be categorized according to the following: material, shape, production process/treatment, origin, surface pattern and grade (Dubin, 2009; Dubin, 1999; Liu, 2012; Liu and Holland, 2017; Sarina, 2022).

The indigenous beads industry notwithstanding has gained recognition as an important economic activity by which many families eke their living. It is on record for generating income for many families (NCC, 2011; Akoto, 2013). It has also been recognized that the cultural tourism segment of the beads industry has become an important source of employment for the local people (Akoto, 2013; Akpabli, 2013). No gainsaying that the potential of traditional/ indigenous knowledge in economic development of local communities associated with beading business is highly recognized all over the world (WIPO, 2010; Iyoro and Ogungbo, 2013), as it is seen as a set of techniques, perceptions, information and behavior that guide local community members to use natural resources (Iyoro and Ogungbo, 2013). This has aroused the interest of some persons particularly in Nigeria to explore bead making from wheat flower. Generally, wheat flour is mainly composed of starches in the form of polymer (i.e. macromolecule). In other words, starch being a polysaccharide (complex carbohydrate) consists of a chain of glucose molecules that are joined together in covalent bonds. It has a basic chemical formula of $(C_6H_{10}O_5)_n$, where n is the number of bonded glucose molecules that grow the chain. For example, a starch having 50 (fifty) glucose molecules in the chain, the formula for such starch would become $(C_6H_{10}O_5)_{50}$. Scheme 1[a] represents the unit chemical structure of wheat flour, while Figure 1[b] is repeated units of glucose, and thus starch as a polymer, which represents the chemical structure of polymeric wheat flour.



[a]



[b]

**Scheme 1:** [a] Unit chemical structure of wheat flour and [b] chemical structure of polymeric wheat flour.

Locally made beads from baking powder, specifically wheat flour, have gained much interest in recent times, particularly from local community artisans in Nigeria as a means of livelihood, as customers especially the poor and the average patronize them for their parties, aphrodisiac reasons, attires and as materials for other artistry works. However, their patronage is hampered seriously as a result of some major draw backs associated with them, which are; (i) undesirable light weight (ii) fragility problem (iii) poor fastness property (iv) unsymmetrical smoothness or irregular and rough surface and (v)

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compromised water resistibility. Moreover, no work has been carried out or reported on the chemical process of bead production from baking powder (wheat flour) with or without the use of composite materials. All of this makes this study novel in the field of material science in particular and, chemistry in general.

This work is thus expected to illustrate the chemistry of wheat flour bead, with the goal of producing a superior bead of quality over the conventional wheat flour beads that are associated with the problems of undesirable light weight, fragility, poor fastness property, irregular and rough surface, as well as compromised water resistibility. This will create a more attractive market for beads made from wheat flour.

## MATERIALS AND METHODS

The equipment used for this study include the following: Infrared spectroscope (IR), electronic weighing balance, flask shaker, oven, Scanning Electron Microscope (SEM), table, knife, palm frond, beaker, conical flask, and plastic bowls. While the chemicals and other materials include commercial wheat flour, plaster-of-paris (POP) cement, titanium dioxide, zinc oxide, barium sulfate, calcium carbonate, cascamite glue and colorant which were locally obtained from Pyrex – IG Science Company Limited, Benin City, Edo State, Nigeria.

### Preparation of the Dough and Bead

Preparation of the dough started with the identification of the best composite material for the bead formulation. Here, the wheat flour and the different composite materials (such as zinc oxide, barium sulphate, titanium dioxide, and calcium carbonate) were weighed with a known amount of 50g each per batch, into a bowl and with and without 20g of POP cement in the mixture using an electronic weighing balance. This was followed by the addition of adhesive (50g cascamite glue and 2ml of red colorant (paint/color mixture). A controlled amount of water was added gradually and mixed thoroughly to a smooth non sticky consistency dough. Thereafter, the dough was needed for 10 - 15 minutes. The *dough was shaped to a known length and size*. Broom stick was used to make a hole in the *bead* while the electronic weighing balance was used to measure the wet weight, and subsequently allowed to dry at room temperature for 24hours. The resulting bead samples were labeled accordingly and characterized physically and chemically.

### Physical Characterization

The physical parameters such as weight, fragility, fastness property, water resistibility, and heat tolerance were analyzed. The following procedures were used in determining the above stated physical properties of the produced beads.

### Weight loss determination

The determination of weight loss using percentage weight loss was carried out using an electronic weighing balance to measure the wet and dry weights of all the bead samples. Initial samples were placed in conical flasks containing water until they attained water adsorption saturation. The wet samples were placed in an oven at 100°C for 4hrs. This was done according to the America Society for Testing and Materials. (ASTM D570). The percentage weight loss (%) was computed using equation (1) (Gao*et al*., 2003) as follows:

$$\text{Weight (\%)} = \frac{W_w - W_d}{W_d} \times 100 \quad (1)$$

where; $W_w$ = Weight of wet sample; $W_d$ = Weight of dry sample.

### Fragility determination

The fragility property of the produced bead samples was carried out with the use of a flask shaker machine, set at varying time (10 – 60mins) and speed (100 – 700m/s). Here, the samples were placed in a conical flask and agitated at varying time and speed until some of the samples broke. The stability of the different samples to the agitated condition was noted to determine which of the samples was fragile and those that were strong.

### Fastness property determination

The determination of colour fastness property of all samples was done practically via the sunlight method, being a direct method for measuring evidently the colour fastness of materials. This was done by placing the samples (stored in a glass cabinet) under sunny atmosphere for one - two weeks. Then colour fading was checked against the original colour. The beads were placed back in the sunny atmosphere for additional one to four months & above period of time, and compared again, to see lightness, and saturation of colour difference/fading. Any sample that did not fade after four months is an indication that that bead sample is very light resistant and can be used under intense display lights for a long period of time.

### Water resistibility determination

The water resistibility test was carried out according to the American Society for Testing and Materials (ASTM D570). Here the conical flask test was used and a calibrated weighing balance was used to measure the water absorption of samples. The procedure involves placing 500ml distilled water into five different conical flasks for a set time (10 – 60 mins) and the amounts of water thus absorbed by the bead samples were determined, after removing them from the conical flasks, they were dried with a clean lint – free cloth and weighed. For all samples, the water uptake was calculated as percentage water absorption, deducible from Equation 1.

### Determination of heat tolerance

Heat tolerance is used to determine the ability of the material to withstand heat, and as such, all samples were subjected to heat using the oven with an optimum time (30mins) but varying temperature to determine the heat resistance of all samples. At the end of the experiment, samples resistibility to different temperature conditions was thus determined.

### Surface analysis

The use of Scanning Electron Microscope (SEM) was used to analyze the surface of the wheat flour, before and after (produced beads), to see how smooth and uniform the surfaces could be. The microstructures of wheat flour particles and the produced bead surfaces were observed by a Scanning Electron Microscope (JSM – 5410LV). The flour sample was analyzed directly in powder form while the produced bead was first freeze dried; the dried bead was then coated with silver (Au) to provide conductivity (Nijdam and Langrish, 2006). The observation distance and contrast of the scanning was adjusted to get the best photographic results, and the microstructure image of each sample was observed at different range of 250x to 1000x magnification.

### Chemical Analysis

Infra – red (IR) spectroscopy was employed in the chemical analysis of the base material, before (wheat flour) and after (bead) the chemical reaction.

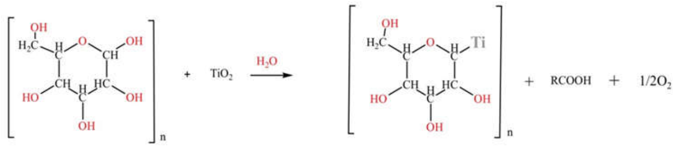
### Infra – red Spectroscopy

The sample was mixed with 0.5ml of ground potassium bromide. This mixture was then placed onto the face of a KBr plate, and the second window was placed on top. With a gentle circular and back and forth rubbing motion of the two windows, the mixture was evenly distributed between the plates, and the mixture then appeared slightly translucent. The sandwiched plates were then placed in the spectrometer. The Fourier transform infrared spectrum was recorded using Agilent FTIR carry 630 spectrometer in the wavelength range 400-4000 $cm^{-1}$ by potassium bromide pellet technique with a resolution and scanning speed of 4 $cm^{-1}$ and 2 mm/sec, respectively.

## RESULTS AND DISCUSSION

### Titanuimation of the wheat polysaccharide

The chemical equation in scheme 2 showed the titaniumation of wheat polysaccharide with titanium dioxide where carboxylic acid and oxygen gas were given off as by-products




**Scheme 2:** The chemical interaction between wheat flour and titanium dioxide that can be referred to as the 'Titaniumation' of wheat flour. The attachment of titanium to the wheat flour was confirmed using the infra-red spectra. Both spectra (Figures 1 and 2) were similar. However, the peak with low intensity appearing around 650 $cm^{-1}$ is attributed to the presence of the titanium in the polysaccharide bead, which is lacking in the untitaniumated polysaccharide wheat flour. Sayed *et al.*, (2016) had attributed a band absorption of 690.52 $cm^{-1}$ to titanium in azo dye that was interacted with titanium dioxide.

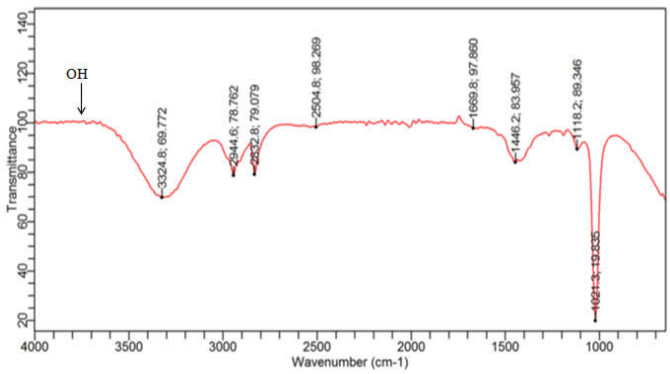



**Figure 1**: The Infra - red spectrum of untitaniumated wheat flour

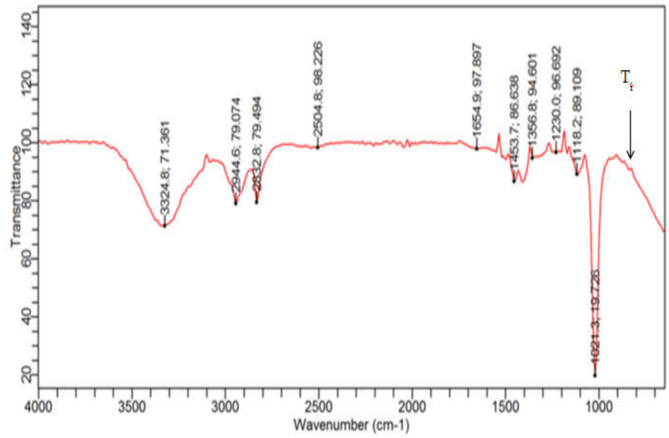



**Figure 2:** The infra - red spectrum of titaniumated wheat flour (produced polysaccharide bead)

The effect of the titanium on the structure of the polysaccharide bead caused stretching and stunting to flattening of the OH band around 3650 – 3750 cm-1, which is expected as the titaniumation of the wheat flour resulted in titanium being substituted into the position of the OH of the first carbon in the ortho position, thereby displacing the OH in that position, which later formed the carboxylic acid as a by-product as seen in the chemical equation. This therefore confirmed the titaniumation of the wheat flower polysaccharide.

## PHYSICAL CHARACTERIZATION

### Weight loss determination

Weight loss determination computed in percentage weight loss was carried out on all the samples of beads that were prepared with the same amount of base materials. Suffice to note that POP used above 20 g resulted in a material not able to hold together as they crumbled away. Result presented in Table 1 showed that after drying, the weight loss per cent of all the composite materials ranged from 0.82 – 9.92%, where the bead made from the wheat flour with the blend of titanium dioxide exhibited the lowest weight loss (0.82%), and the highest weight loss

**Table 1: Weight loss determination of all samples**

| Weight loss determination (from water uptake %) for all Samples | | | |
|---|---|---|---|
| **Samples** | **Wet weight (g)** | **Dry weight (g)** | **Weight (%)** |
| Wheat Flour (WF) | 7.31 | 6.65 | 9.92 |
| ZnO + WF | 8.10 | 7.65 | 5.55 |
| $TiO_2$ + WF | 8.56 | 8.49 | 0.82 |
| $CaCO_3$ + WF | 7.57 | 7.20 | 5.13 |
| $BaSO_4$ + WF | 6.30 | 6.01 | 4.82 |
| $CaCO_3$ + WF + POP | 8.40 | 7.98 | 5.20 |

material was the bead made from the wheat flour without composite material, with weight loss of 9.92%. Thus weight of bead improved with 9.1% with bead made from wheat flower in combination with titanium dioxide as composite over the wheat flower bead without titanium dioxide.

### Fragility determination

The fragility of all the beads that was determined using the flask shaker machine at different times and speeds ranging from 10min/100 $m^{-s}$ to 60min/700$m^{-s}$ have their results presented in Table 2. From the results, all the beads except the bead made from wheat flour in combination with $TiO_2$ broke significantly at 40min/500$m^{-s}$. It implies that the bead made using $TiO_2$ as a composite material with the wheat flour is less fragile, and can be conveniently transported and used without the fear of breaking easily. This advantage is associated with the reinforcement property of $TiO_2$ on the base material because titanium dioxide is known for high toughness, and hardness (Shah *et al*., 2022). Characteristics that have endeared titania (common name for titanium dioxide) for massive use in a variety of both conventional and advanced engineering applications. This is another superior quality exhibited by the bead made from wheat flour using $TiO_2$ as a composite material.

**Table 2: Fragility determination of all samples**

| Fragility of all Samples | | | | |
|---|---|---|---|---|
| **Samples** | **10mins/100$m^{-s}$** | **20mins/300$m^{-s}$** | **40mins/500$m^{-s}$** | **60mins/700$m^{-s}$** |
| Wheat Flour (WF) | No fragility | No fragility | Broken | Broken |
| ZnO + WF | No fragility | No fragility | Broken | Broken |
| $TiO_2$ + WF | No fragility | No fragility | No fragility | Broken |
| $CaCO_3$ + WF | No fragility | No fragility | Broken | Broken |
| $BaSO_4$ + WF | No fragility | No fragility | Broken | Broken |
| $CaCO_3$ + WF + POP | No fragility | No fragility | Broken | Broken |

### Fastness property

The fastness property of all the synthesized beads is shown in Table 3. The fastness property was determined between one week to four months & above period of time. Results showed that they were all stable up to three weeks, hence they were ascribed negative. As at one month, ZnO, $BaSO_3$ and $CaCO_3$ + POP composite beads however showed fading property, hence ascribed positive. This could be associated with oxidation of the bead material, especially the colourant. Extending the period to four months and above, only bead made from $TiO_2$ remained unfaded and thus exhibited excellent resistance to sun radiation over the rest. This can be associated with the effect of the $TiO_2$ that is highly-refractive with UV-resistant property. In another perspective, however, titanium dioxide is semi conductive and exhibits properties capable of inducing the formation of the hydroxyl radical (OH) (Molina-Rayes *et al*., 2020), and as such, one may expect the interaction of UV light with $TiO_2$ to reduce the fastness property due to possible free electrons behavior and electron holes formation in the crystal lattice of $TiO_2$ (George *et al*., 2015). But this effect may have been countered due to the gelatinization and retro gradation of the starch from wheat flour, which subsequently enhanced the binding force between the photochromic colorant and the polysaccharide wheat flour and, thus stabilizing its fastness property. Another important factor why the presence of $TiO_2$ was able to enhance the fastness property of the produced bead is because of its reflective nature. Being a ultra-white inorganic substance (known for being the whitest and brightest pigment), $TiO_2$ was able to exhibit excellent reflective qualities against the UV rays, and thus stabilizing the fastness property of the product. No wonder it is an important raw material for paints and surface coatings. With this entire characteristic, $TiO_2$ is credited with the ability to scatter and absorb UV rays. Again, another possibility of oxidation that could have led to the reduction of the fastness property with $TiO_2$ is the formation of reactive free radical in the presence of oxygen and water (Diebold, 1995; Woditsch and Westerhaus (1993), but this effect was inhibited by the hydrophobic nature of both the polysaccharide and $TiO_2$, as well as the binding force between the photochromic colorant and the polysaccharide wheat flour, which altogether offer stability on the fastness property of the produced bead.

**Table 3: Fastness property of all samples**

| Fastness property of all Samples | | | | | |
|---|---|---|---|---|---|
| **Samples** | **1 week** | **3 weeks** | **1 month** | **3 months** | **4 months & above** |
| Wheat Flour (WF) | Negative | Negative | Negative | Positive | Positive |
| ZnO + WF | Negative | Negative | Positive | Positive | Positive |
| $TiO_2$ + WF | Negative | Negative | Negative | Negative | Negative |
| $CaCO_3$ + WF | Negative | Negative | Negative | Negative | Positive |
| $BaSO_3$ + WF | Negative | Negative | Positive | Positive | Positive |
| $CaCO_3$+ WF+POP | Negative | Negative | Positive | Positive | Positive |

**Key:** Negative means no fading while positive means fading

### Water resistibility

The water resistibility of the produced bead was studied between the period of 10 - 60 mins via their respective water adsorptive capabilities. Results as presented in Table 4 showed that all the beads were resistant between the period of 0 – 10 mins. However, as from 15 – 60 mins (1 hr) where they showed water absorption saturation, water absorption (%) was found to range between 0.75 – 4.08%, and bead made from $TiO_2$ exhibited the least water absorption while the conventional wheat flour bead exhibited the highest water absorption, thus making titanium based bead to be of superior water resistant. The almost insignificant (0.75%) water uptake by the titaniumated bead may be ascribed to the presence of residual materials. Otherwise, both the base material and $TiO_2$ are hydrophobic in nature. This implies that titaniumated beads have no significant problem when they come in contact with water.

**Table 4: Water resistance of all samples**

| | Water absorption (resistibility) at different times | | | | | |
|---|---|---|---|---|---|---|
| **Samples** | **10min** | **15min** | **25min** | **30min** | **60min** | **Absorption** |
| Wheat Flour (WF) | NA | 7.54 | 7.68 | 7.83 | 7.83 | 4.08 % |
| ZnO + WF | NA | 7.68 | 7.74 | 7.83 | 7.84 | 2.42% |
| $TiO_2$ + WF | NA | 8.00 | 8.02 | 8.04 | 8.04 | 0.75% |
| $CaCO_3$ + WF | NA | 7.10 | 7.14 | 7.16 | 7.18 | 1.98% |
| $BaSO_3$ + WF | NA | 6.03 | 6.08 | 6.13 | 6.15 | 2.27% |
| $CaCO_3$+ + WF + POP | NA | 7.89 | 7.90 | 7.97 | 8.00 | 2.00% |

**Key:** NA means no absorption. Initial weights are; Wheat flour = 7.51 g, ZnO+ WF = 7.65 g, $TiO_2$+ WF = 7.98, $CaCO_3$+ WF = 7.04 g, $BaSO_3$+ WF = 6.01, $CaCO_3$+ WF + POP = 7.84 g.

## Heat tolerance

The heat tolerance of all the produced beads were determined and results as presented in Table 5 showed that all beads were heat tolerant up to 50$^o$C except $BaSO_3$ based bead. While ZnO based bead was able to tolerate heat up to 100$^o$C, only $TiO_2$ based bead was able to tolerate 125$^o$C heat and, thus described as heat resistant. This can be attributed to the reinforcement from the $TiO_2$ composite material with a very high melting point of 1843$^o$C. A unique thermal characteristic that qualifies it to be sought after as a material for advanced applications in areas such as photo protection, solar thermal energy cultivation, heat relieving, heat transportation, heat storage, etc. (Shah *et al*., 2022).

**Table 5: Heat tolerance of all samples**

| | Temperature ($^o$C)/ Time (Mins.) | | | | |
|---|---|---|---|---|---|
| **Samples** | **24oC 30mins** | **30oC 30mins** | **50oC 30mins** | **100oc 30mins** | **125oc 30mins** |
| Wheat Flour (WF) | Positive | Positive | Positive | Negative | Negative |
| ZnO + WF | Positive | Positive | Positive | Positive | Negative |
| TiO2 + WF | Positive | Positive | Positive | Positive | Positive |
| CaCO3 + WF | Positive | Positive | Positive | Negative | Negative |
| BaSO3 + WF | Positive | Positive | Negative | Negative | Negative |
| CaCO3+WF+POP | Positive | Positive | Positive | Negative | Negative |

**Key:** Positive means able to withstand heat while negative means not able to withstand heat.

## Surface morphological study

The Scanning Electron Microscope (SEM) images of the wheat flour (before) and the locally produced polysaccharide bead from wheat flour that was titaniumated using $TiO_2$ are presented in Figure 3 [a] & [b]. Figure 3[a] revealed the wheat endosperm lumps, which formed some kind of unsymmetrical shapes. Wheat endosperm however, includes mainly starch granules and protein.

[a] [b]

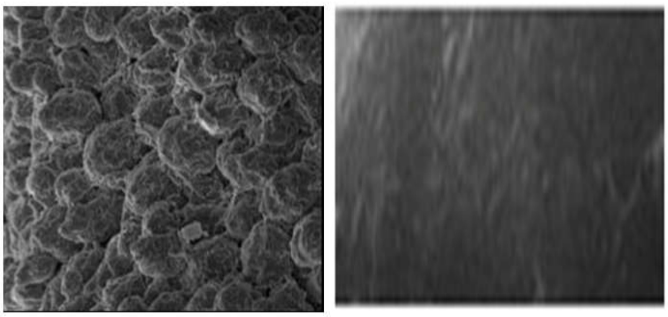


**Figure 3:** [a] Surface image of wheat flour and [b] surface image of produced bead.

A few studies have been carried out on the surface chemical composition of wheat flour (Lin *et al*., 2019; Berton *et al*., 2002).

Although it has been demonstrated that the surface chemical composition of flour is closely related to water absorption and hydration during the formation of the dough (Berton *et al*., 2002), but the influence of surface chemical composition on flour quality is still not clear. Comparing both SEM images, it is obvious that the titaniumation of the wheat flour significantly improved the surface morphology of the resulting bead, i.e. from a rough and unsymmetrical surface to a smooth and symmetrical surface. This could be the reason why the wheat flour bead without composite absorbs the highest water, and in comparison with the titanium based bead, it is >5 fold difference. The photographic image of the produced bead is shown in Figure 4.

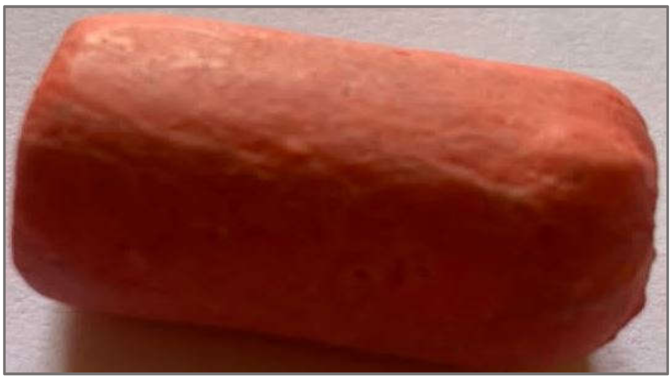


**Figure 4:** Photo image of titaniumated wheat flour bead.

## CONCLUSION

The study on both the preliminary selection and determination of material for producing a superior quality bead over the conventional bead made from just wheat flour proved successful. This study showed that bead made from wheat flour in the blend of $TiO_2$as a composite material significantly resulted in improved quality bead over the conventional wheat flour bead. Superiority in terms of improved weight, less fragility, better fastness property, and excellent water resistant and heat tolerance were achieved. The introduction of the titanium dioxide into the wheat flour in the presence of water gave a successful chemical interaction (as supported by the FTIR results), which resulted in the improved quality of the produced bead. This work, has therefore, been able to show a novelty of the chemistry of polysaccharide wheat flour bead using titanium dioxide as a composite material.

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