IN THIS ISSUE



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IMAGE: OBSERVATORY WITH STAR TRAILS ON MOUNTAINS FOR CLEAR SKY

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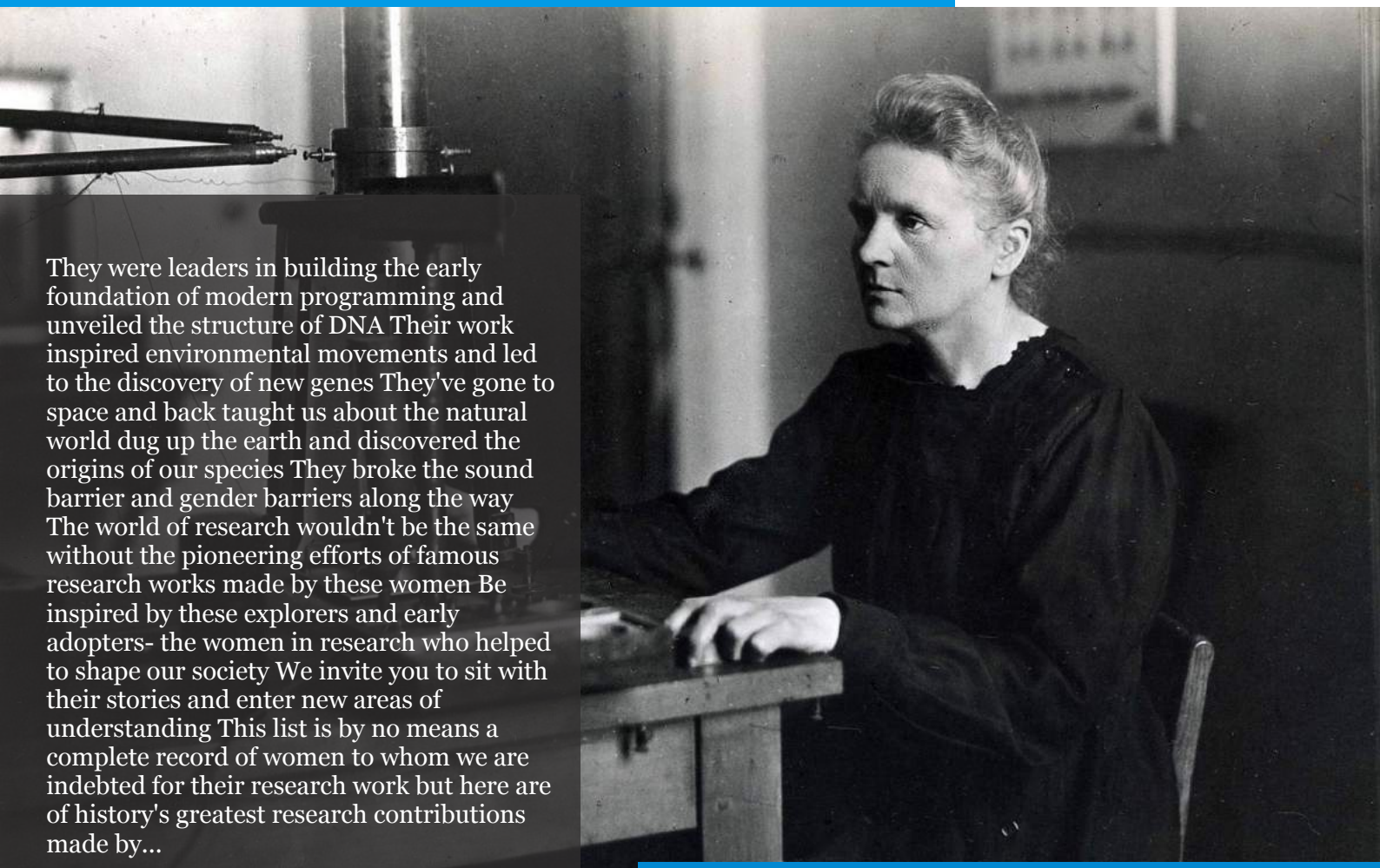

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Research papers and articles

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# Population Growth as a Biological Factor in Climate Change

*Dimitrios Kampolis*

*Hellenic Open University*

## ABSTRACT

Climate change is a concept that is directly correlated with everyday life. Extreme weather events are occurring with greater intensity and frequency, most likely caused by an increase in the Earth's average temperature. Dozens of studies have been based on the anthropogenic contribution of the increase in greenhouse gases, which are identified as the main cause of global warming. But what is the contribution of the human species itself as a biological factor in this increase?.

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# Population Growth as a Biological Factor in Climate Change

Dimitrios Kampolis

## ABSTRACT

*Climate change is a concept that is directly correlated with everyday life. Extreme weather events are occurring with greater intensity and frequency, most likely caused by an increase in the Earth's average temperature. Dozens of studies have been based on the anthropogenic contribution of the increase in greenhouse gases, which are identified as the main cause of global warming. But what is the contribution of the human species itself as a biological factor in this increase?*

*Author:* School of Applied Arts and Sustainable Design, Hellenic Open University.

## I. INTRODUCTION

A few years ago, in a BBC documentary for the bunkers of World War II in the English Channel, southeast England, a question was asked about the heating of the space and its source. Guide's answer was disarming, naming the only source of heat as the presence of thousands of people inside the bunker. Bunker's system is a small closed system in which, however, the biological presence of human had left its thermal footprint.

The purpose of this study is to prove the possible contribution of global population to a much larger closed system, that of earth's atmosphere, in temperature increase and in atmospheric composition.

### *1.1 Methodology*

#### *1.1.Human Body*

Human body produces heat through metabolic activities. An average human body consumes 2000 Calories per day (1 Cal = 4,184 × 103 joules) which is equal to 8,37 × 106 Joules per day. Since most of this energy escapes body as heat, it implies that an average body emits 348.000 J/hr which is equal to ~100Watts [1] [2].

Therefore, the energy produced by a human in a year is:

$$8.37\times106 \times 365 \text{ days} = 3{,}055\times109 \text{ J/yr} \quad (1)$$

#### *1.1.2 Conversion of Energy Into Temperature*

To convert energy to temperature it is sufficient to divide it by the mass of the heating medium (atmospheric air in kg) and divide the result by the Cp of the air which is ~1006 J/kg oC.

Since 1kg of atmosphere requires 1006 J to rise T = 1 degree Celsius, the total mass of the atmosphere requires:

$$5{,}181\times1021 \text{ J to rise T} = 1 \text{ degree Celsius} \quad (2)$$

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The mass of atmosphere is:

$$\sim 5{,}15\times1018 \text{ kg} \tag{3}$$

(1),(2) result to the annual thermal footprint of a human body in Earth's atmosphere:

$$0{,}589\times10\text{-}12 \text{ degrees Celsius} \tag{4}$$

### *1.2 Worldpopulation growth*

World population is increasing during last decades [5].

1960: 3,035×109

1970: 3,7×109

1980: 4,458×109

1990: 5.327×109

2000: 6,143×109

2010: 6,956×109

The population growth from 1960 to 2010 (fig.1) is:

$$3{,}921\times109 \text{ persons} \tag{5}$$

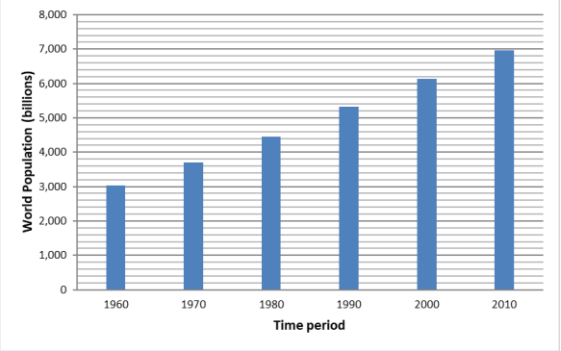


*Fig. 1:* Global population growth from 1960 2010.

### *1.3 Global Temperature Increase*

Global temperature is increasing last decades with a temperature increase of ~0.65 degrees Celsius (1960: ~0.0 degrees Celsius, 2010: ~0.65 degrees Celsius (fig.2).

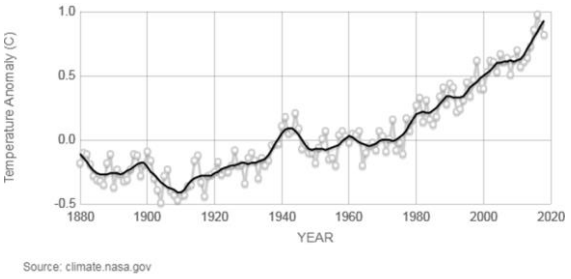


*Fig. 2:* Global temperature increase from 1890-2018.

Therefore from (1),(4),(5) we calculate the contribution of world population over a period of 50 years in temperature increase which is:

$$0{,}58\times10\text{-}1 \text{ degrees Celsius} \sim 0.06 \text{ degrees Celsius} \quad (6)$$

This value constitutes a minimum ~9% of the total temperature increase during period 1950-2010 and by reduction to actual daily action conditions [6] (~170Watts), this contribution reaches a ~15%

### *1.4 Atmosphere*

Human body consumes and produces several gases.

#### *1.4.1 O2*

The average human breath is ~7,5lt air/min [3] resulting to:

$$3{,}942\times106 \text{ lt/yr} \quad (7)$$

21% of this volume is O2 resulting to:

$$0{,}828\times106 \text{ lt/yr} \quad (8)$$

During a breath 3-6% of O2 is retained -we assume an average of around 4% (9)

(7),(9) result to the volume of O2 removed from atmosphere by the respiratory system of an average human:

$$0{,}033\times106 \text{ lt/yr} \quad (10)$$

Therefore from (5),(10) we calculate the volume of O2 removed from atmosphere by the respiratory system of population growth over 50 years:

$$3{,}24\times1015 \text{ lt} \quad (11)$$

$$\text{One lt of O2 weights } 1{,}43\times10\text{-}3 \text{ Kg} \quad (12)$$

(11), (12) result to the mass of O2 removed from atmosphere by the respiratory system of population growth over 50 years:

$$4{,}64\times1012 \text{ kg} \quad (13)$$

According to measurements [7], the natural decrease in O2 is 4ppm per year (Fig.3) which results to:

$$5{,}64\times10\text{-}9 \text{ kg/l} \quad (14)$$

(13), (14) result to the mass of O2 removed from atmosphere due to natural reasons over 50 years:

$$1{,}452 \text{ x}1015 \text{ kg} \tag{15}$$

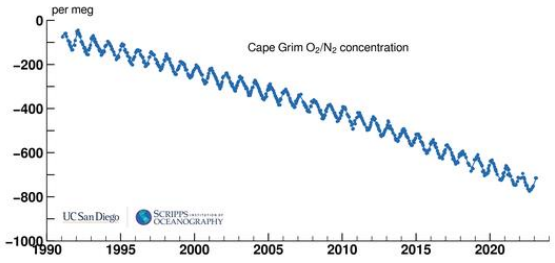


*Fig.3:* O2 decrease from 1990-2023

### *1.4.3 CH4*

An average human produces about 0,35lt/day [4] which results to:

$$127{,}75\text{lt/yr} \tag{16}$$

(5), (16) result to the volume of CH4 produced by the population growth over 50 years:

$$250{,}4\times109 \text{ lt} \tag{17}$$

$$\text{One lt of CH4 weighs } 0{,}6682\times10\text{-}3 \text{ Kg} \tag{18}$$

(17), (18) result to the mass of CH4 produced by the population growth over 50 years:

$$0{,}167\times109 \text{ kg} \tag{19}$$

Cows produce about 250-500lt/day (average=325lt) resulting to:

$$0{,}118\text{x}106 \text{ lt/yr} \tag{20}$$

The population of cows is

$$1{,}5\times 109 \tag{21}$$

(18),(20),(21) result to the mass of CH4 produced by the cow population over 50 years:

$$0{,}119\times1015 \text{ kg} \tag{22}$$

### *1.4.4 Atmospheric pressure and sea level*

Average Pressure is 1013.25hPa and it is correlated to the mass of atmosphere (5,15 × 1018 kg).

Human respiratory system has removed mass of O2 affecting MSL pressure in an order of 0.9x10-3 hPa over the last 50 years while natural O2 decrease has affected MSL pressure in an order of 0.28hPa.

Human digestive system has produced mass of CH4 affecting MSL pressure in an order of 0,033x10-6 hPa over the last 50 years while CH4 increase due to cow population has affected MSL pressure in an order of 0.023hPa.

In total the change in MSL pressure is ~0.258hPa (23)

1hPa atmospheric change = 10mm of sea level change

## II. RESULTS

The contribution of world population in temperature increase is ~+0.06-0.150C and to the composition of atmosphere due to human respiration is -4.64×1012Kg of O2 and +16.73×106 Kg of CH4 (50 years period) (Table 1).

*Table 1:* Results

| | Heat (°C) | $O^2$ (Kg) | $CH_4$ (Kg) |
|---|---|---|---|
| Human | +0.06-0.12 | $-4.64x10^{12}$ | $+0.167x10^{9}$ |
| Total | +0.65 | $-1.452,5\ x10^{12}$ | $+118,8 \times 10^{12}$ |

## III. CONCLUSIONS

The study results to a strong contribution of world population to temperature increase (9-15% of the total temperature increase during the period 1960-2010) while the contribution in O2 declination is very small (0.3% of current declination), and in CH4 increase is negligible (comparing to current increase).

Further work is needed to investigate the contribution of O2 and CH4 in changes of mean atmospheric pressure and mean sea level.

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# Development a Soluble Antioxidant Beverage of Tree Tomato (*Solanum Betacea*) for Sportsmen

*Fernando Buitrón & Jenny Ruales*

## ABSTRACT

Athletes require specific hydration that contains antioxidants in addition to carbohydrates and electrolytes, because the tree tomato (Solanum betaceum) is rich in antioxidants, the objective of the present study was the elaboration of a hydrating drink in tree tomato powder for athletes. The pulp was chemically and nutritionally characterized and then treated with an enzymatic cocktail to increase dissolved solids. To define the pulp spray drying conditions, the following variables were tested: feed flow (12 and 17 mL/min); drying temperature (130, 140, 150 and 180 °C); maltodextrin concentration (3, 7 and 9%) and the best drying condition was selected by sensory analysis. The fruit was processed and formulated of the hydrating drink powder, in which solubility, process effectiveness, sensory characteristics and polyphenol content were analyzed. The optimum drying conditions were drying temperature of 140 °C, feed flow of 12 ml/min, and addition of maltodextrin at 9%; tricalcium phosphate was added at 100 ppm to improve effectiveness. The dehydrated pulp contained a high content of β-carotenes, minerals, polyphenols and antioxidants. The product formulation contained 37.47% pulp providing 120 mg of polyphenols per-100 g of product. It was packed in a 40 g trilaminate to prepare 600 mL. Finally, the product stability study (16.7°C and 67-80% relative humidity) for three months showed a shelf life of one year.

*Keywords:* solanum betaceum, moisturizing powder, antioxidant, sportsmen, spray drying.

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# Development a Soluble Antioxidant Beverage of Tree Tomato (*Solanum Betacea*) for Sportsmen

Desarrollo de una bebida soluble antioxidante con base en tomate de árbol (*Solanum betacea*) para deportistas

Fernando Buitrón[α*] & Jenny Ruales[σ]


## RESUMEN

*Los deportistas requieren hidratación que además de carbohidratos y electrolitos contenga antioxidantes y dado que el tomate de árbol (Solanum betacea) rica en antioxidantes el objetivo del presente estudio fue la elaboración de una bebida hidratante en polvo de tomate de árbol para deportistas. Se realizó la caracterización química y nutricional de la pulpa que luego fue tratada con un cóctel enzimático para incrementar los sólidos disueltos. Para definir las condiciones de secado de la pulpa por aspersión se probaron las variables: flujo de alimentación (12 y 17 mL/min); temperatura de secado (130, 140 150 y 180 °C); concentración de maltodextrina (3, 7 y 9%) y la mejor condición de secado se seleccionó con un análisis sensorial. Se procesó la fruta para realizar la formulación de la bebida hidratante en polvo, en la que se analizó la solubilidad, efectividad del proceso, características sensoriales y contenido de polifenoles. Las condiciones óptimas de secado fueron temperatura de secado de 140 °C, flujo de alimentación de 12 mL/min, y adición de maltodextrina al 9%, se adiciona fosfato tricálcico a 100 ppm para mejorar la efectividad. La pulpa deshidratada tuvo un alto contenido de β-carotenos, minerales, polifenoles y antioxidantes. La formulación del producto tuvo un 37,47% de pulpa que proporciona 120 mg de polifenoles por cada 100 g de producto. Se empacó en un trilaminado de 40 g para preparar 600 mL. Finalmente, el estudio de estabilidad del producto (16,7°C y 67-80% de humedad relativa) durante tres meses mostró una vida útil de un año.*


*Palabras Clave:* solanum betacea, hidratante en polvo, antioxidante, deportistas, secado por aspersión.


## ABSTRACT

*Athletes require specific hydration that contains antioxidants in addition to carbohydrates and electrolytes, because the tree tomato (Solanum betaceum) is rich in antioxidants, the objective of the present study was the elaboration of a hydrating drink in tree tomato powder for athletes. The pulp was chemically and nutritionally characterized and then treated with an enzymatic cocktail to increase dissolved solids. To define the pulp spray drying conditions, the following variables were tested: feed flow (12 and 17 mL/min); drying temperature (130, 140, 150 and 180 °C); maltodextrin concentration (3, 7 and 9%) and the best drying condition was selected by sensory analysis. The fruit was processed and formulated of the hydrating drink powder, in which solubility, process effectiveness, sensory characteristics and polyphenol content were analyzed. The optimum drying conditions were drying temperature of 140 °C, feed flow of 12 ml/min, and addition of maltodextrin at 9%; tricalcium phosphate was added at 100 ppm to improve effectiveness. The dehydrated pulp contained a high content of β-carotenes, minerals, polyphenols and antioxidants. The product formulation contained 37.47% pulp providing 120 mg of polyphenols per-100 g of product. It was packed in a 40 g trilaminate to prepare 600 mL. Finally, the product stability study (16.7°C and 67-80% relative humidity) for three months showed a shelf life of one year.*


*Keywords:* solanum betaceum, moisturizing powder, antioxidant, sportsmen, spray drying.

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## I. INTRODUCCIÓN

Los deportistas de alto rendimiento realizan un esfuerzo físico que genera pérdida de electrolitos por medio de la sudoración que causa acumulación de ácido láctico, daño muscular, deshidratación y producción de radicales libres que provocan problemas degenerativos, por ello la necesidad de la hidratación con una bebida que no sólo contenga electrolitos y carbohidratos para restaurar las pérdidas de dichos componentes sino también antioxidantes y polifenoles para neutralizar el efecto de los radicales libres (Urdampilleta et al., 2015).

Los principales antioxidantes contenidos en frutas y vegetales tienen la capacidad de prevenir enfermedades crónicas no transmisibles asociadas al estrés oxidativo. Es así que la pulpa de *Solanum betacea* (tamarillo o tomate de árbol) se destaca por presentar vitamina C, vitamina E, provitamina A, minerales (potasio, calcio, cobre, hierro, manganeso y magnesio) y compuestos antioxidantes, como antocianinas y carotenoides (Alves et al., 2017). Sin embargo, en el país no se ha explotado el potencial de esta fruta para la hidratación de deportistas.

El tomate de árbol (*C. betacea*) es una planta nativa de América del Sur Bolivia, Chile, Ecuador, y Perú. *S. betacea* fue domesticada en el norte de Perú y el sur de Ecuador; y comercialmente se lo cultiva en Colombia, Ecuador, Perú y en Nueva Zelanda (Alves et al., 2017). En Ecuador *S. betacea* se cultiva en un rango de 2000 a 3000 msnm., en las provincias de Carchi, Imbabura, Pichincha, Cotopaxi, Tungurahua, Chimborazo, Bolívar, Cañar, Azuay y Loja (Revelo Moran et al., 2004), donde podemos encontrar cuatro genotipos: rojo punton, anaranjado gigante, amarillo punton y mora. Existen en mayor cantidad los genotipos anaranjado y amarillo y en menor cantidad los morados (Chamba Vaca, 2018).

Según Morillas-Ruiz & Delgado-Alarcón (2012), *S. betacea* tiene una concentración de compuestos fenólicos 2010,40 ± 0,02 µgGA/g (µg de ácido gálico por gramo de fruta) que le confieren capacidad de inhibición de radicales libres y actividad antioxidante, y además posee actividad prebiótica ya que se ha identificado hidrocoloides (proteína arabinogalactana) y polisacáridos hemi celulósicos productores de ácidos grasos de cadena corta. Por lo tanto, *S. betacea* es un alimento funcional potencial debido a sus propiedades biológicas, efectos antioxidantes, antiinflamatorios, antivirales, antibacterianos, anti depresivos, anti cancerígenos y sus pigmentos naturales se asocian con la prevención de enfermedades crónicas.

Debido a las características que presenta *S. betacea* y atendiendo a la necesidad de los deportistas de alto rendimiento, el principal objetivo de esta investigación es elaborar una base deshidratada de pulpa de tomate de árbol de genotipo anaranjado gigante para la preparación de una bebida hidratante.

## II. MATERIALES Y MÉTODOS

### *2.1 Obtención y preparación de la materia prima.*

El tomate de árbol (*S. betaceum*) de genotipo anaranjado gigante se obtuvo en el cantón Cevallos, provincia de Tungurahua. Se utilizaron 80 kg de fruta madura, previamente seleccionada con la escala de color 5-6 de la norma técnica NTE INEN 1 909:2015 (INEN, 2015).

Para obtener la pulpa, se lavó la fruta con agua potable y cloro con una concentración de 50 ppm, se cortaron los pedúnculos y la cáscara se retiró mediante pelado manual, luego se trocea la fruta con el desintegrador RIETZ de 5 HP modelo RP-8-k-115, marca General Electric. El despulpado de la fruta se

realizó en el pulpatador Langenkamp modelo M5707 con una malla de poro N° 23 para separar las pepas de la pulpa. La pulpa obtenida y los desechos se pesaron para calcular el rendimiento del proceso. Finalmente, se empacó la pulpa en bolsas de polietileno de 8 kg y fueron almacenadas a -19 °C hasta su utilización.

### *2.2 Caracterización fisicoquímica de la pulpa de S. betacea*

La caracterización fisicoquímica de la materia prima consistió en evaluar los sólidos solubles totales, pH, índice de solubilidad (ISA) y materia insoluble en alcohol (MIA). El contenido de sólidos solubles totales fue determinado con la medición de grados Brix de cada muestra al colocar 2 gotas de pulpa de tomate de árbol en un brixómetro Hand Held Refractometer CHASE modelo Nº 80-109. El pH se determinó en muestras de 40 mL de pulpa homogenizada en un vaso de precipitación de 250 mL con un pH-metro ORION modelo 21 OA calibrado antes de cada medición.

Para la determinación de la materia insoluble en alcohol (MIA) se colocaron 20 g de pulpa (M) homogenizada dentro un tubo Falcon y se centrifugó durante 10 minutos a 5000 rpm, concluido luego se separó el sobrenadante. A la pulpa residual, se agregó 20 mL de alcohol etílico al 90 %, se agitó empleando un vortex y luego se centrifugó por 10 minutos a 4000 rpm. El proceso se repitió hasta que la pulpa se tornó de color blanco. La pulpa blanca fue depositada en una caja Petri y se registró el peso inicial (P1), luego fue sometida a secado en una estufa a 70 °C durante 24 horas. Una vez enfriada la caja Petri en un desecador se registró el peso final (P2) y se calculó el porcentaje de MIA con la Ecuación 1.

$$\% \, MIA = \frac{P_2 - P_1}{M} \times 100 \qquad \text{[Ec. 1]}$$

### *2.3 Caracterización nutricional y de compuestos bioactivos de la pulpa de S. betacea.*

La caracterización nutricional y de compuestos bioactivos consistió en los análisis: proximal, vitaminas, minerales, azúcares, ácidos orgánicos, polifenoles y la capacidad antioxidante.

El análisis proximal de la pulpa de *S. betacea* consistió en la determinación de la humedad, extracto etéreo, proteína, ceniza y carbohidratos. La humedad se determinó según el método descrito en AOAC (2007), 920.151, (37.1.12) utilizando una estufa de vacío a 70 °C y 100 mm de Hg. El Extracto Etéreo (E.E) fue determinado según el método descrito en AOAC (2007), 934.06, (37.1.10) y 920.39, (4.5.01), mediante secado en estufa de vacío y extracción con éter etílico o de petróleo en un equipo Goldfisch. El contenido de Proteína (P) se terminó según el método explicado en AOAC, (2007), 920.152, (37.1.35). El contenido de cenizas se determinó según el método descrito en AOAC, (2007), 940.26, (37.1.18), en una mufla a 525 °C. El contenido de carbohidratos (C) se determinó según el método señalado por Hart y Fisher (1991). Para determinar el valor energético se utilizó la Ecuación 2 (Hart y Fisher, 1991).

$$Valor\ energético \left[\frac{kcal}{100\ g}\right] = (P \times 4) + (E.E \times 9) + (C \times 4) \qquad \text{[Ec. 2]}$$

El contenido de Vitamina C como de β-carotenos se determinó con el método descrito por (DECAB, 2004b). Este análisis se realizó en un equipo de HPLC marca Hewlett Packard (HP), modelo 1050 series, usando un detector de UV-VIS.

El análisis de contenido de minerales (calcio, magnesio, sodio, potasio, zinc, manganeso, hierro y cobre) en la pulpa de tomate de árbol realizó por espectroscopia de absorción atómica según el método DECAB-01(Ruales et al., 2000).

El contenido de ácidos orgánicos se cuantificó siguiendo el método descrito por Pérez et al. (1997). Mientras que el contenido de azúcares se cuantificó aplicando el método Modificado del Manual de la columna ASTEC NH2 (DECAB, 2004a).

La extracción y determinación de polifenoles solubles totales se realizó según el método descrito por Slinkard & Singleton (1977) y la capacidad antioxidante, se determinó aplicando el método ABTS indicado por Re et al. (1999).

Hidrólisis de la pulpa de *S. betacea*.

Con el objeto de disminuir la carga de sólidos insolubles (SIS) y aumentar la cantidad de sólidos disueltos (SD) se realizó la hidrólisis de la pulpa. Para esto, se descongeló la pulpa e hidrolizó a 30°C durante 30 minutos adicionando 1 mL/kg de cóctel enzimático Rap Vegetable Juice®.

### *2.4 Secado Por Aspersión de la Pulpa de S. Betacea*

El proceso de secado por aspersión se realizó en el equipo de secado marca BUCHI-B299, modelo MM-LAN 045 con una temperatura de salida entre 80 – 100 °C. La pulpa fue mezclada con maltodextrina para mejorar el proceso de secado e incrementar el rendimiento y la solubilidad del producto seco (MIRAVET VALERO, 2009). Previo a definir las condiciones del proceso de secado, probaron 13 tratamientos con los factores i) flujo de alimentación con dos niveles (12 y 17 mL/min); ii) temperatura de secado con cuatro niveles (130, 140 150 y 180 °C); iii) concentración de maltodextrina con tres niveles (3, 7 y 9%).

*Efectividad del proceso*

La efectividad del proceso se evaluó por la cantidad de sólidos disueltos en la pulpa (Ecuación 3). Donde EF corresponde al porcentaje de efectividad, SD a los sólidos disueltos que incluyen la adición de maltodextrina y el 3% de Sólidos Insolubles. Los SIS corresponden a los sólidos insolubles tomados después de la enzimación

$$\%EF = \frac{Peso\ del\ producto\ final}{Peso\ de\ SD + Peso\ de\ SIS} \times 100 \qquad \text{[Ec. 3]}$$

A partir de los resultados obtenidos en la efectividad del proceso se seleccionaron los tratamientos con mayor rendimiento para la evaluación sensorial de los tratamientos y escoger la formulación más aceptable.

### *2.5 Características sensoriales de la pulpa deshidratada.*

La evaluación sensorial se realizó con un grupo de 10 panelistas semi entrenados a quienes se presentaron las muestras preparadas en forma de bebidas. Cada muestra fue identificada mediante cifras al azar de 3 dígitos. La bebida a evaluar se preparó al mezclar 20 g de la pulpa deshidratada de tomate de árbol (en polvo) con 300 mL de agua de cada tratamiento. Los atributos evaluados fueron el aroma y sabor intenso a tomate de árbol, apariencia y consistencia de la mezcla, presencia de grumos, color y presencia de sabores extraños. Los atributos de sabor y aroma se calificaron usando una recta de 10 cm, los demás atributos se calificaron cualitativamente (Anzaldúa, 1994). El tratamiento que presentó las mejores puntuaciones sensoriales fue seleccionado para formular el polvo hidratante.

### *2.6 Formulación del polvo hidratante*

Una vez determinado el mejor tratamiento de acuerdo con los análisis sensoriales, se procedió a la formulación del producto terminado. Se preparó una mezcla con 10 g de sacarosa, 14 g glucosa y 17 g pulpa deshidratada para preparar 500 mL de bebida. Para la adición de minerales se consideró como referencia algunos productos hidratantes del mercado y como aditivos se añadió fosfato tricálcico

antiaglomerante, sorbato de potasio como preservante, ácido cítrico como acidulante, vitamina C como antioxidante, cloruro de magnesio y cloruro de sodio como electrolitos funcionales y carboximetilcelulosa (CMC) al 1% como modificador de textura. El producto terminado en forma de hidratante en polvo fue empacado en presentaciones de 40 g en bolsa trilaminadas de LDPE/foil/LDPE de 0,043 mm de espesor.

### *2.7 Estabilidad del polvo hidratante.*

Para determinar la estabilidad del producto se evaluó el polvo hidratante durante períodos de 0, 15, 30, 60 y 90 días a temperatura ambiente (18 °C). Al cabo de cada periodo, en cada muestra se determinó el contenido de humedad, ISA. También se realizó un análisis sensorial al día 0 y 75 para determinar la vida útil del producto. Para comprobar la inocuidad del producto final se realizó el análisis microbiológico por recuento del total de aerobios y de hongos y levaduras (INEN, 2014) .

Para la determinación del IAA, ISA y PH se siguió el método de Anderson et al. (1969), con algunas modificaciones. Se pesaron 1,25 g (b.s) de harina en un tubo de centrífuga previamente pesado, se adiciona 30 mL de agua destilada y se colocó en baño maría a 60 °C durante 30 min con agitación constante en un baño termostático (Lab Companion BW-20H) equipada con una plancha de agitación sumergible (Variomag). Se dejó enfriar a temperatura ambiente para llevar a centrifugación, a 4900 rpm durante 30 minutos en una centrífuga (Bioblock Scientific Sigma 2-15). Luego, se decantó el sobrenadante, se midió su volumen y se tomó una alícuota de 10 mL en una caja Petri previamente pesada y se secó en estufa a 70 °C durante 24 horas. Posteriormente, se pesó nuevamente. El gel retenido en los tubos se pesó. El IAA, ISA y PH se determinaron de acuerdo a las ecuaciones 5 a 7, respectivamente (Rodríguez, Lascano, Sandoval, 2012).

El contenido de humedad se determinó por el método gravimétrico 920.151.(37.1.12) (AOAC, 2007), para lo cual se colocaron 2 g de polvo hidratante en cajas Petri y se llevó a una estufa de vacío a 70 °C hasta sequedad.

### *2.8 Análisis estadístico*

La distribución normal de la eficiencia del proceso fue verificada con la prueba de Shapiro-Wilks con un nivel de significancia de 0,05. Se realizó un análisis de factores simples entre las variables temperatura, concentración de maltodextrina y flujo de alimentación con la variable eficiencia del proceso para determinar las mejores condiciones de secado. Todos los análisis se realizaron con el software SigmaStat y la herramienta Statwin.

## III. RESULTADOS

### *3.1 Caracterización nutricional y de compuestos bioactivos de la pulpa de S. betacea.*

La pulpa de tomate de árbol presentó valores de sólidos totales de 10 grados Brix, y la media (con las desviaciones típicas entre paréntesis) de pH fue de 3,6 (0,02) y el porcentaje de MIA de 1,26 (0,07). Se observó que los sólidos totales de la pulpa hidrolizada incrementaron a 12 grados Brix, mientras que el pH disminuyó ligeramente a 3,46 (0,02).

La caracterización nutricional se encuentra expresada en valores en base seca (BS) (Tabla 1), se evidenció que la pulpa de tomate de árbol contiene altas concentraciones de carbohidratos, vitamina C, y minerales como el potasio, magnesio y calcio. Las concentraciones de polifenoles y antioxidantes fueron altas tanto en la pulpa como en la pulpa deshidratada (Tabla 5).

*Tabla 1:* Caracterización nutricional de la pulpa de *S. betacea* (n=3). (Media de los parámetros y su desviación estándar entre paréntesis)

| Parámetro | Unidades/100 g BS | Media |
|---|---|---|
| Humedad | % p/p | 84,02 (1,09) |
| Proteína | g/100 g | 14,85 (0,23) |
| Grasas | g/100 g | 4,49 (0,25) |
| Carbohidratos totales | g/100 g | 75,63 (0,51) |
| Valor calórico | Kcal/100 g | 367,73 |
| Vitamina C | mg/100 g | 105,00 (9,00) |
| B-carotenos | mg/100 g | 22,90 (1,50) |
| Azúcares | | |
| Fructosa | g/100 g | 10,14 (0,76) |
| Glucosa | g/100 g | 10,64 (0,15) |
| Sacarosa | g/100 g | 12,13 (0,89) |

LND : Límites no detectables
BS: Base seca

*Tabla 1:* Caracterización nutricional de la pulpa de *S. betacea* (n=3). Continuación…(Media de los parámetros y su desviación estándar entre paréntesis)

| Componente | Unidades/100 g BS | Media |
|---|---|---|
| Ácidos orgánicos | | |
| Ácido cítrico | g/100 g | 15,41 (0,33) |
| Ácido tartárico | g/100 g | LND |
| Ácido málico | g/100 g | 1,97 (0,15) |
| Minerales | | |
| Calcio | mg/100 g | 160 (5,20) |
| Magnesio | mg/100 g | 97,85 (2,90) |
| Sodio | mg/100 g | 0,40 (0,01) |
| Potasio | mg/100 g | 2490,69 (71,00) |
| Zinc | mg/100 g | 1,20 (0,10) |
| Hierro | g/100 g | 1,30 (0,01) |
| Cobre | g/100 g | 0,50 (0,03) |

LND : Límites no detectables
BS: Base seca

*Selección del tratamiento en base a la efectividad del proceso y las características sensoriales de la pulpa deshidratada*

A partir de la evaluación de la efectividad del proceso (Tabla 2), se seleccionaron los tratamientos que presentaron efectividades superiores al 35% y corresponden a los tratamientos T3, T12, T7, T5, T11, T9 y T2 a los cuales se realizó el análisis sensorial (Tabla 3). Se descartó el T13, ya que a esta temperatura la consistencia del producto era pastosa y no era apta para el producto final.

*Tabla 2:* Evaluación de la efectividad del proceso

| Tratamiento | Temperatura (°C) | Flujo (mL/min) | Maltodextrina (g/kg de pulpa) | Efectividad del proceso (%) |
|---|---|---|---|---|
| T1 | 180 | 12 | 30 | 18,43 |
| T2 | 180 | 17 | 70 | 34,83 |
| T3 | 180 | 12 | 70 | 43,71 |
| T4 | 180 | 17 | 30 | 14,62 |
| T5 | 170 | 12 | 90 | 38,01 |
| T6 | 165 | 14,5 | 70 | 28,2 |
| T7 | 150 | 17 | 70 | 39,11 |
| T8 | 150 | 12 | 70 | 26,84 |
| T9 | 150 | 17 | 30 | 36,57 |
| T10 | 150 | 12 | 30 | 27,22 |
| T11 | 150 | 12 | 90 | 37,04 |
| T12 | 140 | 12 | 90 | 42,00 |
| T13 | 130 | 12 | 90 | 35,48 |

Al evaluar el efecto del flujo de alimentación sobre la efectividad del proceso, los tratamientos seleccionados no presentaron diferencias significativas ($p > 0,05$), siendo el flujo de alimentación de 12 mL/min, el que presentó la mejor efectividad del proceso. Tampoco se presentaron diferencias significativas entre los tratamientos al evaluar el efecto de la concentración de maltodextrina sobre la efectividad del proceso ($p > 0,05$). En este caso, la tendencia fue que la concentración de maltodextrina de 90 g/kg de pulpa presentó las mejores eficiencias del proceso. Finalmente, al evaluar la temperatura y la efectividad del proceso tampoco se presentaron diferencias significativas ($p > 0,05$), y la tendencia mostró que la efectividad del proceso mejora a 140 y 150 °C de temperatura, mientras que las temperaturas de 150 y 180 °C provocaron que parte del producto se queme.

*Tabla 3:* Puntuaciones del Análisis sensorial de los tratamientos seleccionados

| Tratamiento | Condiciones °C/(mL/min)/(g/kg) | Aroma (U) | Sabor (U) | Color (U) | Aspecto (U) | Sabores extraños (U) |
|---|---|---|---|---|---|---|
| T9 | 150/17/30 | 4,01 | 4,26 | 4,68 | 4,60 | 5,80 |
| T7 | 150/17/70 | 2,93 | 2,51 | 5,85 | 3,35 | 5,56 |
| T3 | 180/12/70 | 4,18 | 3,20 | 7,35 | 5,51 | 6,90 |
| T2 | 180/17/70 | 3,93 | 3,01 | 7,26 | 4,51 | 5,48 |
| T12 | 140/12/90 | 8’00 | 8’64 | 7’86 | 8’57 | 0’00 |
| T11 | 150/12/90 | 7,79 | 7,29 | 7,43 | 8,54 | 2,00 |

Aunque los tratamientos seleccionados presentaron las mejores eficiencias de proceso, durante el análisis sensorial (Tabla 3), se presentaron altas puntuaciones en el parámetro de sabores extraños (quemado) para los tratamientos T9, T7, T3 y T2. Y los tratamientos T11 y T12 obtuvieron las mayores puntuaciones para los parámetros de aroma, sabor, color y aspecto, se seleccionó al T12 (140 °C, flujo de 12 mL/min y una concentración de maltodextrina de 90 g/kg) como el mejor tratamiento por la ausencia de sabores extraños.

Una vez seleccionadas las condiciones del proceso de secado, se mejoró la efectividad del mismo al 50% con la adición de 100 ppm fosfato tricálcico de para obtener un mejor arrastre y menos compactación en las paredes del atomizador (MIRAVET VALERO, 2009).

### *3.2 Formulación del polvo hidratante*

La formulación del polvo hidratante a partir de la pulpa deshidratada a 140 °C, flujo de 12 mL/min, una concentración de maltodextrina de 90 g/kg y 100 ppm de fosfato tricálcico se presenta en la Tabla 4. En esta formulación fue necesario añadir aditivos para que el polvo hidratante cumpla con los requerimientos de azúcares y minerales de una bebida hidratante. Además, se adiciona carboximetilcelulosa (CMC) para mejorar la estabilidad del producto disuelto, fosfato tricálcico (anticompactante) para aumentar la solubilidad y sorbato de potasio como conservante del producto final. El valor calórico del producto final fue de 119,79 calorías en 600 mL.

*Tabla 4:* Formulación de la bebida hidratante en polvo

| Ingredientes | Porcentaje (%) |
|---|---|
| Pulpa deshidratada | 37,47 |
| Azúcar | 25,10 |
| Glucosa | 35,59 |
| Fosfato tricálcico | 0,37 |
| Estabilizante CMC | 0,37 |
| Cloruro de sodio | 0,56 |
| Ácido cítrico | 0,11 |
| Ácido ascórbico | 0,11 |
| Cloruro de magnesio | 0,15 |
| Sorbato de potasio | 0,15 |
| Total | 100 |

Aunque la concentración de sales minerales, polifenoles y antioxidantes disminuyó en el producto final, aún cumple con los requerimientos de una bebida hidratante para actividad deportiva (Tabla 5).

*Tabla 5:* Comparación de nutrientes de interés entre la pulpa deshidratada y el polvo hidratante. (Media de los parámetros y su desviación estándar entre paréntesis)

| Parámetro | Unidades | n | Pulpa Deshidratada | n | Polvo hidratante |
|---|---|---|---|---|---|
| Polifenoles | mg/100g muestra | 3 | 223,65 (6,44) | 3 | 115,23 (4,29) |
| Capacidad Antioxidante | µmol/100g muestra | 3 | 929,88 (55,35) | 3 | 513,84 (10,62) |
| Cloruro de sodio | g/100g | 3 | | 1 | 0,62 |
| Ca | mg/kg | 3 | 160 (5,20) | 1 | 6,72 |
| Na | mg/kg | 3 | 0,40 (0,01) | 1 | 136,00 |
| Mg | mg/kg | 3 | 2490,69 (71,00) | 1 | 150,60 |
| Zn | mg/kg | 3 | 1,20 (0,10) | 1 | LD |

LD: Límite de detección

Por otro lado, se puede evidenciar la inocuidad del producto final, puesto que en el contaje total de aerobios la muestra no presenta crecimiento de bacterias ni levaduras, mientras que presentó el conteo mínimo de hongos (Tabla 6).

*Tabla 6:* Análisis microbiológico producto terminado

| Parámetro | Conteo UFC/g |
|---|---|
| Contaje Aerobios | 10 [(a)] |
| Contaje Hongos | $2,6 \times 10^3$ |
| Contaje Levaduras | 10 [(b)] |

*UFC. Unidades Formadoras de colônias.*

a. *Estimado de Aerobios Contaje en Placa, fuera de rango 25-250, en los análisis la muestra en la dilución $10^{-1}$, no hay crecimiento de bacterias.*
b. *Estimado de levaduras Contaje en Placa, fuera de rango 10-150, en los análisis la muestra en la dilución $10^{-1}$, no hay crecimiento de levaduras.*

### *3.3 Estabilidad del polvo hidratante*

Se determinó que el punto crítico de humedad para el producto final es del 9% de una muestra compactada y el punto crítico de solubilidad de la pulpa deshidratada es del 24%. Como se observa en

la Figura 1, la humedad del producto final no alcanza al punto crítico mientras que la solubilidad supera el punto crítico durante el periodo evaluado.

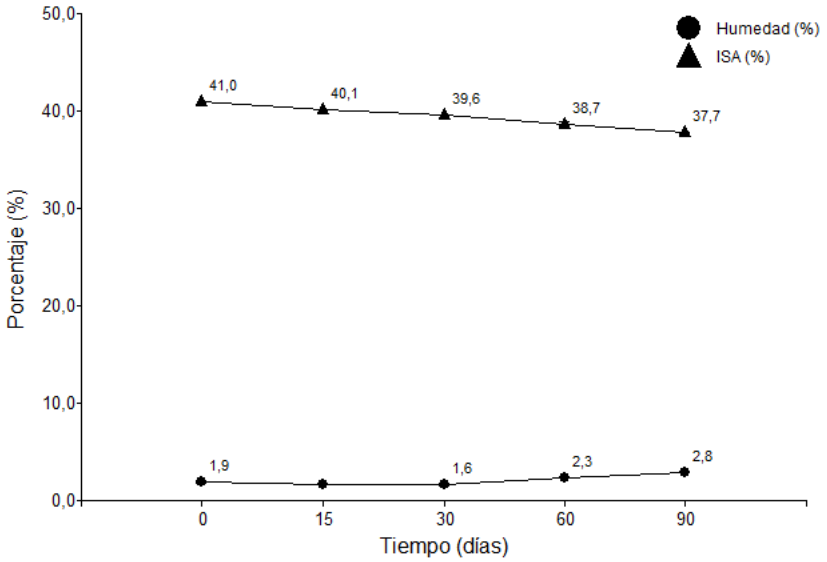


*Figura 1:* Evolución del porcentaje ISA y el porcentaje de humedad medidos en los 0, 15, 30, 60 y 90 días

Las ecuaciones de la vida útil del polvo hidratante de acuerdo con el porcentaje de ISA y la humedad se visualizan en la Tabla 7 Sin embargo, de acuerdo con el análisis se determinó que la vida útil del producto es de 330 días almacenado en percha a una temperatura ambiente de 16,7 °C y humedad relativa entre el 67 y 80%.

*Tabla 7:* Vida útil del polvo hidratante

| Parámetro | Ecuación | Límites Críticos | Días a límites críticos |
|---|---|---|---|
| Solubilidad | y = -0,7248x + 41,509 | 24 | 360 |
| Humedad | y = 0,257x + 1,3008 | 9 | 450 |

## IV. DISCUSIÓN

Los contenidos de ácido cítrico y ácido málico, B-carotenos y polifenoles de la pulpa de *S. betacea* fueron superiores a los valores reportados por Acosta-Quezada et al. (2015) en cultivares ecuatorianos de *S. betacea* anaranjado y se mantuvieron dentro de los rangos el contenido de azúcares (fructosa, glucosa y sacarosa). En El caso de los polifenoles, el valor se asemeja al valor encontrado en la jalea de semillas de *S. betacea* anaranjado (Acosta-Quezada et al., 2015). Mientras que los valores de B-carotenoides encontrados en este estudio concuerdan con los presentados por Ali Hassan & Abu Bakar (2013) en cultivares de *S. betacea* morado de Malasia. Las diferencias presentadas pueden deberse a que los contenidos dependen de varios factores como la variedad, estacionalidad, condiciones de cultivo, almacenamiento, etc.(Márquez et al., 2017).

Los rendimientos obtenidos en la presente investigación, asemejan a los de Siddick & Ganesh (2016), quienes encontraron una temperatura de 164 °C como la óptima para la recuperación de pulpa deshidratada de *C. betacea*, por otro lado se encontró que el secado a 140 °C la solubilidad de los polvos de *C. betacea* se incrementa en comparación a los polvos secados en temperaturas inferiores y el mayor

rendimiento a una temperatura de secado de 120 °C (Herrera Campos, 2018). Este último resultado difiere con nuestros resultados ya que a 130 °C se obtuvo un polvo de consistencia pastosa, esto podría deberse a las diferentes proporciones de maltodextrina utilizadas en los ensayos y la no adición de goma arábiga. Ya que tanto la maltodextrina y la goma arábiga se utilizan como portadores de material para eliminar la pegajosidad y aumentar la temperatura de transición vítrea de la mezcla, sin embargo, se debe tomar en cuenta que la aplicación excesiva de maltodextrina reduce la aceptación del consumidor (Moghbel et al., 2019). Por otro lado, en futuros procesos para mejorar los rendimientos del secado por aspersión se puede implementar otro tipo de portadores de material como proteínas, polisacáridos (pectina) y surfactantes (Moghbel et al., 2019).

Al realizar el proceso de secado por aspersión se contribuyó a mantener las actividades antioxidantes de *S. betacea*, como lo demuestran Dillwyn et al. (2022), quienes midieron las mayores actividades antioxidantes para el proceso de secado por aspersión en comparación con el secado al sol, secado en bandejas. El polvo hidrante fue formulado para suplir las calorías necesarias para restaurar una hora de práctica deportiva, ya que la falta de hidratación en los deportistas conlleva efectos negativos, especialmente si la actividad física es de larga duración. Según Urdampilleta et al. (2013) una deshidratación superior al 2% incrementa la frecuencia cardiaca y la temperatura corporal hasta los 40 °C, lo que obliga al deportista a detener la actividad física. Por lo tanto, los deportistas deben ingerir líquidos con una frecuencia concreta de volúmenes y concentraciones de electrolitos adecuados. En cuanto al contenido de electrolitos de la bebida hidratante de *S. betacea*, la formulación se realizó en base a los minerales establecidos en las etiquetas de los hidratantes en el mercado debido a que cumplen con las recomendaciones para bebidas de deportistas de 450-700 mg/L de sodio (Olivos et al., 2012). Aunque aún no es un requisito la adición de compuestos funcionales para la hidratación de deportistas (Feye, 2018), la bebida hidratante de *S. betacea* formulada en el presente estudio posee un valor nutricional superior a las bebidas hidratantes comerciales debido a su alto contenido de vitaminas y antioxidantes debido a que ayudan a combatir el aumento de la producción de radicales libres y estrés oxidativo (Olivos et al., 2012).

El producto final cumple cabalmente con los requerimientos de carbohidratos y electrolitos para un deportista y además tiene propiedades funcionales por la presencia de polifenoles. El contenido de polifenoles y la capacidad antioxidante determinados en el producto final es alto y de acuerdo con el equivalente de Trolox, de *S. betacea,* esta bebida es una fuente intermedia de antioxidantes (Márquez et al., 2017), el potencial antioxidante de este producto hidratante es su contenido de 120 mg de polifenoles por cada 100 g de producto. El contenido de ácido ascórbico del producto final es inferior al obtenido por Castro et al. (2022) en una bebida similar y se debió que existió una degradación total de la vitamina C por el proceso térmico y se añadió vitamina C. La pulpa deshidratada del producto final también es un buen aporte de minerales, que de acuerdo con Torres (2012), *S. betacea* presenta buena bioaccesibilidad de Ca y Fe.

El análisis microbiológico del producto demuestra que no hay crecimiento de bacterias ni levaduras y el conteo de hongos está dentro del límite permisible, esto se debería a la presencia de compuestos fenólicos con posibles características antimicrobianas (Zhao et al., 2009). Mientras que la evaluación de la estabilidad del producto final indica que en un empaque trilaminado es posible almacenarlo hasta un año sin que pierda sus características organolépticas. El producto final es competitivo dentro del mercado, porque hasta el momento no se han desarrollado bebidas hidratantes naturales y con antioxidantes y polifenoles, que coadyuvan en la hidratación y a combatir enfermedades cardiovasculares.

## V. CONCLUSIONES

La pulpa deshidratada tuvo una correcta rehidratación y sin presencia de sabores extraños; el spray dryer en el proceso de secado existía compactación en las paredes del deshidratador, requirió la adición de anticompactante, para evitar la compactación de polvo en las paredes del mismo y tener mayor rendimiento.

El proceso se definió con las siguientes condiciones 12 mL/min de flujo de alimentación, 140 ºC de temperatura de entrada de aire a la cámara, adición de maltodextrina al 9% y la adición de 100 ppm de anticompactante, que resultó en un incremento del rendimiento del proceso del 50% y el incremento del 29% de solubilidad.

El producto final resultó en un hidratante natural y funcional con el aprovechamiento de las propiedades antioxidantes y electrolitos que posee *S. betacea* (tomate de árbol), las propiedades antioxidantes se mantuvieron con el proceso de secado por aspersión, sin embargo fue necesario fortalecer la bebida con vitamina C, obteniendo un producto ideal para la reposición de energía y electrolitos perdidos en la práctica deportiva, que es comparable con las bebidas energéticas del mercado con contenido superior en polifenoles.

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# Calculation of Sediment Accumulation Volume in Aparan Reservoir

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## ABSTRACT

Long-term and efficient operation of a reservoir largely depends on the correct solution of problems considering such principal aspects as the forecast of fluid and solid river flows, taking into account climatic and anthropogenic factors; the evaluation of the amount of sediment deposited in the reservoir; the prediction of the possible accidents scenario and assessment of their consequences. The purpose of this paper is to determine the volume of eventually accumulated soil mass in a reservoir. As an example, the calculations are given for the Aaparan Reservoir located in the Republic of Armenia. The study has been based on the initial hydrometric data on the flow rate and sediment load, the design characteristics of the reservoir. Three principal methods have been chosen for the calculated estimation of the deposited sediment volume. Field measurements in the Aparan reservoir were carried out using precise geodetic means. Comparison of the obtained calculation results and field measurements enables to assess the reliability of the calculation methods, and to reveal the inaccuracies of a number of initial data. Despite the significant discrepancy between the obtained values of the deposited silt volume, the recorded maximum value does not exceed 1.5% of the reservoir useful volume. The calculation results make it possible to arrive at a conclusion that over half a century of operation, the design parameters of the Aparan reservoir remain practically unchanged. This proves that the Kasakh River, which flows into the reservoir, has a very weakly expressed alluvial regime.

*Keywords:* reservoir, stream, hydrometric data, silt, sediment, flow, useful volume, dead volume.

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## ABSTRACT

*Long-term and efficient operation of a reservoir largely depends on the correct solution of problems considering such principal aspects as the forecast of fluid and solid river flows, taking into account climatic and anthropogenic factors; the evaluation of the amount of sediment deposited in the reservoir; the prediction of the possible accidents scenario and assessment of their consequences. The purpose of this paper is to determine the volume of eventually accumulated soil mass in a reservoir. As an example, the calculations are given for the Aaparan Reservoir located in the Republic of Armenia. The study has been based on the initial hydrometric data on the flow rate and sediment load, the design characteristics of the reservoir. Three principal methods have been chosen for the calculated estimation of the deposited sediment volume. Field measurements in the Aparan reservoir were carried out using precise geodetic means. Comparison of the obtained calculation results and field measurements enables to assess the reliability of the calculation methods, and to reveal the inaccuracies of a number of initial data. Despite the significant discrepancy between the obtained values of the deposited silt volume, the recorded maximum value does not exceed 1.5% of the reservoir useful volume. The calculation results make it possible to arrive at a conclusion that over half a century of operation, the design parameters of the Aparan reservoir remain practically unchanged. This proves that the Kasakh River, which flows into the reservoir, has a very weakly expressed alluvial regime.*

*Keywords:* reservoir, stream, hydrometric data, silt, sediment, flow, useful volume, dead volume.

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## I. INTRODUCTION

Reservoirs are actually unique public infrastructures for storing and regulating river flows. They occupy a special place among water bodies and support people's social and economic activities by supplying water and controlling floods. On the other hand, reservoirs make their important contribution to ensuring the security of national economy. The construction and operation of reservoirs have a long history, however, some issues of long-term, safe and efficient operation of many structures are still insufficiently studied. Here we can note the need for the following studies:

- reliable and long-term forecasting of fluid and solid river water flows' dynamics related to significant changes in both climatic and ecological conditions, and anthropogenic impacts [Doll P. & Muller (2012), Tokmajyan H.V. (2016), Dobrovolsky S.G. (2011), Vardanyan T.G. (2007)],
- periodic assessment of the decreasing useful volume of the reservoir, due to sedimentation processes occurring there and, in accordance with this, develop a prospect planning and repair program

designed to clean up deposits or reduce the amount of silt in the stream [Baljyan P.H. & Balյyan V.P. (2013), Baljyan P.H. & Sedrakyan S. M. & Manukyan A. S. (June, 2014), SP 58.13330.2012 (2003)],

- determination of characteristics of possible washout and destruction of earth dams, and forecasting the boundaries of the wave flow propagation [Prudovsky A.M. (1998), Zhang H. & Youssef H. & Long H.D. & Kahawita R.A. (1992)].

During operation, sediment, carried away by water, wind or ice, is accumulated in the reservoir. Generally, in the bottom part of a reservoir, a certain "dead" volume is normatively specified for sedimentation (Fig. 1). However, the practice of operation of small and medium-sized reservoirs, built in mountain-foothill zones shows that a small part of silts - mainly suspended particles, is often deposited in their dead volumes [Baljyan P.H. et al. (June, 2014), Davranov G.T. & Fyrlina G.L. (2017), Kamara Usman (1993)]. In many cases, the form of sedimentation is more dependent on the reservoir drawdown schedule. Two options are possible here:

1. During the year, the water mark is at the banked-up water level (BWL);
2. Before the spring flood, the useful volume of the reservoir is used, and the water mark comes down to the dead volume level (DVL).

In the first case, the entrained sediment and coarse fraction of suspended particles move downstream by the flow and settle down to the bottom of the useful volume of the reservoir (Fig. 1).

In the second case, we have the following picture: during the spring floods, when the reservoir begins to fill, the front of sediment deposition from the lower elevations begins to rise upstream to the BWL. In the summer dry season, a considerable part of accumulated water is supplied for household needs and technological activities, as a result the water level in the reservoir is gradually lowered, exposing the upper sections of the deposits forming small islands. The river flow here begins to wash away a small part of the deposited sediment and move them to the lower levels. When the useful volume of the reservoir is fully used, the sediment begins to fill the dead volume. The described process is repeated in subsequent years. In both cases, the main part of the deposited sediment occupies the useful volume, thereby gradually decreasing the regulating capacity of the reservoir (Fig. 1)

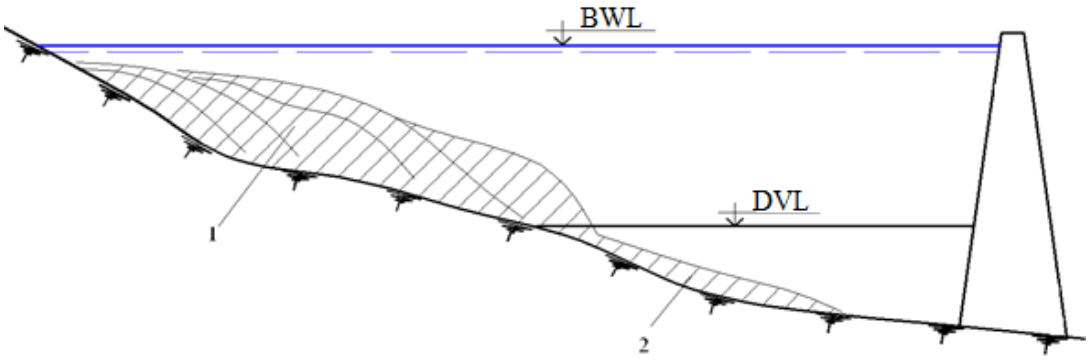


*Fig. 1:* Schematic sketch of the typical pattern of the longitudinal sediment deposition in the reservoir during operation.

*1- deposits of larger sediment particles in a useful, regulating volume,*
*2- deposits of small particles in the dead volume*

There are frequent cases when, due to an incorrect forecast of the alluvial regime of a waterway, within three to four decades, most of the reservoir basin is filled with soil mass and as a result the structure is practically becomes out of order. The foregoing enables to come to a conclusion that the study of the

sediment regime of the waterway and the calculation of the volume of sediment deposited in the reservoir is of great scientific and practical importance, including planning of various cleaning operations.

Therefore, for the long-term and effective operation of reservoirs, the following tasks are of very important scientific and practical importance for the national economy:

- study of the alluvial regime of the watercourse,
- calculation of the volume of sediments deposited in the reservoir,
- development of engineering measures to reduce the amount of sediment flowing into the reservoir,
- planning and carrying out work on cleaning sediment deposited in the reservoir.

There are about seven dozen small and medium-sized reservoirs with earth dams in operation in the Republic of Armenia. The present paper presents research results carried out in the Aparan Reservoir, built from 1962 to 1966 on the Kasakh River. The catchment area of the river at the dam site is 591 km². The Aparan Reservoir, with a total volume of 91 million m³ and a useful volume of 81 million m³, is one of the ten largest reservoirs in the Republic of Armenia. The height of the dam is 53 m, the length along the ridge is 200 m. The operation of the Aparan Reservoir has significantly reduced the flow of water from Lake Sevan for irrigation purposes.

The purpose of the present paper is to determine the volume of sediment deposited in the Aparan Reservoir on the basis of hydrological calculations and field measurements.

This is the first time such studies are carried out in reservoirs of A rmenia. The obtained research methods and results can be used to study other reservoirs of the Republic of Armenia.

Theoretical basis

## II. PROBLEM STATEMENT AND METHODOLOGY

River sediment is of importance in the process of soil mass deposition in the reservoir, both in a suspended state and moving in the lower layers of the flow (entrained sediment). Consequently, the total amount of sediment is due to the alluvial characteristics of the waterway on which a structure is installed. Having sufficiently reliable data on sediment flow and its granulometric parameters, it is possible to build a model and set a mathematical problem to determine the volume of sediment in the reservoir and solve it. In field studies, the design topographic material of the reservoir bowl was used. The research objects are the water and alluvial regimes of the Kasakh River, the original bottom of the Aparan Reservoir and the bed load. Accordingly, the subject of this study is the construction of a mathematical model designed for determination of sediment deposits in the reservoir by the incoming river flow. Collected hydrometric data of the average monthly water and sediment discharge were processed by hydrological methods, Field measurements were carried out in the reservoir and 2D and 3D models of sediment along the length of the reservoir were developed.

The study was carried out within the framework of the program of the research base laboratory "Hydraulic engineering" NPUA with the financial support of the RA MESCS Science Committee.

## III. RESEARCH RESULTS

In calculating the volume of sediments bedded in the Aparan Reservoir, the hydrometric data of the Kasakh River were used. In order to obtain more reliable results, these hydrological calculations were carried out using three methods:

- with direct use of measured hydrometric data on sediment discharge;
- using the values of the sediment discharge calculated from the fluid flow of the Kasakh River;
- based on data from regional maps of river water turbidity.

At the stage of acquiring the initial material, it turned out that the water-flow record of average monthly and average annual discharges of suspended sediments of the Kasakh River is very short. At the same time, there are no measurements of the entrained sediment. Contrary to this, to establish the fluid discharge of the river during 1966 -2018 sufficiently detailed hydrometric measurements were carried out. There is also a regional map on the distribution of the average turbidity of waterways, compiled on the basis of processing the results of extensive hydrological studies [Surface water resources of the USSR, vol. 9, no. 2. Chapter 10 - Bassein r. Araks (1976)].

*Method one*. To calculate the volume of the sediment, we used the data on the discharge of suspended sediments measured at the measuring station "Vardenis", installed on the bed of the Kasakh River, a few kilometers above the Aparan Reservoir. In the section of the measuring station, the catchment area of the river is 441 km², and in the section of the dam - 591 km². The values of the solid discharge measured at the measuring station were transferred to the section of the reservoir by means of hydrological recalculation (Table 1).The tabulated data show that the average annual flow rate is 0.36 kg/s.

*Table 1:* Average monthly flow of sediment of the Kasakh River in the section of Aparan Reservoir, calculated using the same flow rates of the Vardenis measuring station, (kg/s)

| Year | Month | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 |
| 1976 | 0.14 | 0.18 | 0.06 | 0.008 | 0 | 0 | 0,001 | 0,001 |
| 1977 | 0.11 | 0.06 | 0.018 | 0.002 | 0 | 0 | 0 | 0 |
| 1978 | 0.09 | 0.24 | 0.017 | 0.003 | 0 | 0 | 0 | 0 |
| 1979 | 0.02 | 0.01 | 0.02 | 0.004 | 0,003 | 0.002 | 0.001 | 0.001 |
| 1980 | 2,25 | 0.16 | 0.024 | 0.003 | 0.006 | 0.005 | 0.005 | 0.001 |
| Maximum | 11,6 | 14,7 | 0.41 | 0.58 | 0.10 | 0.029 | 0.030 | 0.01 |
| Monthly average | 1.47 | 1,21 | 0.099 | 0.053 | 0.015 | 0.009 | 0.006 | 0.004 |
| Average annual flow of sediment from 1976 to 1980 . - 0.36 kg/s | | | | | | | | |

During the period of the reservoir operation, the volume of sediment inflow is calculated by the ratio

$$W = \frac{Q_S T}{\rho_S} \tag{1}$$

where $Q_S$ is the sediment flow (kg/s); $T$ is the estimated time interval (s); $\rho_S$ is the density of sediment deposits (kg/m³).

*Method two*. In this case, in determining the sediment volume, the values of the sediment flow will be used, calculated for the liquid flow of the Kasakh River. It should be noted that there was a fairly long hydrological record of river discharge. The values of the fluid flow, measured at the Vardenis measuring station, were transferred by recalculation to the section of the reservoir (Table 2). Based on the analysis of a large number of field measurements for the waterways of the Eastern Transcaucasia, a below empiric regularity is proposed that connects the flow of suspended sediment and water [Surface water resources of the USSR, vol. 9, no. 2. Chapter 10 - Bassein r. Araks (1976)]

$$Q_S = 0{,}72\, Q^{0{,}95} \quad (2)$$

Taking into account Eq. (2) for the values of Table 2, the corresponding values of sediment were calculated (Table 3). In this case, the average annual flow rate is 0.95 kg/s. This value can be used to establish the flow rate of the sediment and that part of the suspended sediment, which can be bedded in the reservoir.

The total volume of sediment deposited over 54 years in the Aparan Reservoir in the second method is calculated similarly to the first. The result is:

$$W = \left(\frac{0{,}4 \times 0{,}95}{1400} + \frac{0{,}46 \times 0{,}95}{2200}\right) 54 \times 31{,}54 \times 10^6 = 0,\ 80 \text{ million. m}^3$$

*Table 2:* Average annual flow rates in the section of the Aparan Reservoir recalculated according to fluid flow rates of the Vardenis measuring station

| Year | Water flow, m³/s | Year | Water flow, m³/s | Year | Water flow, m³/s |
|---|---|---|---|---|---|
| 1966 | 2.71 | 1984 | 1.35 | 2002 | 1.52 |
| 1967 | 2.75 | 1985 | 0.49 | 2003 | 1.31 |
| 1968 | 4.00 | 1986 | 0.88 | 2004 | 1.51 |
| 1969 | 3.67 | 1987 | 1.82 | 2005 | 2.47 |
| 1970 | 2.65 | 1988 | 2.76 | 2006 | 2.39 |
| 1971 | 2.60 | 1989 | 0.71 | 2007 | 1.70 |
| 1972 | 4.10 | 1990 | 2.58 | 2008 | 1.13 |
| 1973 | 1.63 | 1991 | 1.17 | 2009 | 0.95 |
| 1974 | 1.51 | 1992 | 1.30 | 2010 | 1.40 |
| 1975 | 2.24 | 1993 | 0.83 | 2011 | 1.64 |
| 1976 | 2.77 | 1994 | 0.92 | 2012 | 1.04 |
| 1977 | 1.26 | 1995 | 0.70 | 2013 | 1.29 |
| 1978 | 3.30 | 1996 | 0.78 | 2014 | 0.76 |
| 1979 | 1.09 | 1997 | 0.91 | 2015 | 1.13 |
| 1980 | 2.04 | 1998 | 0.63 | 2016 | 1.37 |
| 1981 | 0.85 | 1999 | 0.98 | 2017 | 1.04 |
| 1982 | 0.91 | 2000 | 0.82 | 2018 | 0.98 |
| 1983 | 1.03 | 2001 | 0.75 | | |

*Table 3:* Average annual flow discharge in the section of the Aparan reservoir, calculated by fluid flows of the Kasakh River

| Year | Sediment flow, kg/s | Year | Sediment flow, kg/s | Year | Sediment flow, kg/s |
|---|---|---|---|---|---|
| 1966 | 1.86 | 1984 | 0.96 | 2002 | 1.07 |
| 1967 | 1.88 | 1985 | 0.37 | 2003 | 0.93 |
| 1968 | 2.69 | 1986 | 0.63 | 2004 | 1.07 |
| 1969 | 2.47 | 1987 | 1.27 | 2005 | 1.70 |
| 1970 | 1.82 | 1988 | 1.89 | 2006 | 1.64 |
| 1971 | 1.78 | 1989 | 0.52 | 2007 | 1.19 |

| | | | | | |
|---|---|---|---|---|---|
| 1972 | 2.75 | 1990 | 1.77 | 2008 | 0.81 |
| 1973 | 1.14 | 1991 | 0.84 | 2009 | 0.69 |
| 1974 | 1.07 | 1992 | 0.92 | 2010 | 0.99 |
| 1975 | 1.55 | 1993 | 0.60 | 2011 | 1.15 |
| 1976 | 1.89 | 1994 | 0.66 | 2012 | 0.74 |
| 1977 | 0.89 | 1995 | 0.51 | 2013 | 0.92 |
| 1978 | 2.24 | 1996 | 0.57 | 2014 | 0.56 |
| 1979 | 0.78 | 1997 | 0.66 | 2015 | 0.81 |
| 1980 | 1.42 | 1998 | 0.46 | 2016 | 0.97 |
| 1981 | 0.62 | 1999 | 0.71 | 2017 | 0.75 |
| 1982 | 0.66 | 2000 | 0.60 | 2018 | 0.70 |
| 1983 | 0.74 | 2001 | 0.55 | 2019 | - |
| Average annual value of suspended sediment flow | | | | - | 0,95 kg/s |

Method three. According to the regional map the basin of the Kasakh River is located on the territory of which the turbidity of waterways averages 0.3 kg/m³. Taking into account the values of the Table 2 liquid runoff (volume) of the river in the period 1966 to 2020 turns out to be equal to 2.74 billion m³. Then, with the average value of the turbidity of the flow, the total mass of suspended sediment for 54 years will be equal to: $W = 2{,}74 \times 10^9 \times 0{,}3 = 0{,}82$ billion kg or 0.52 million m3. Moreover, 40% of this volume is deposited in the reservoir, i.e. 0.21 million m³.

## IV. RESULTS OF THE STUDY

*Method one:* Studies carried out by this method show that particles with a diameter less than 0.05 mm are transitory and do not deposit in water bodies. Their share is up to 60% of the total amount of suspended sediment [Surface water resources of the USSR, vol. 9, no. 2. Chapter 10 - Bassein r. Araks (1976)]. At the same time, for the waterways of the Araks River, the ratio of suspended and entrained loads has been established. The average amount of entrained sediment, accounting for 46% of the total suspended sediment volume [Ter-Minasyan R.O. (September, 1978)]. Thus, according to the empiric dependence of Eq.(1) for the period of operation of the Aparan Reservoir (1966 to 2020), the total volume of sediment deposition in the first variant will be $Q_S = 0{,}36$ kg/s

$$W = \left(\frac{0{,}4 \times 0{,}36}{1400} + \frac{0{,}46 \times 0{,}36}{2200}\right) 54 \times 31{,}54 \times 10^6 = 0{,}31$$ million m³

where the denominators indicate the values of the densities of the suspended and entrained particles, respectively.

*Method two:* The total volume of sediment deposited in the Aparan Reservoir over 54 years, where $Q_S = 0{,}95$ kg/s, is calculated similarly to the first method. As a result we get

$$W = \left(\frac{0{,}4 \times 0{,}95}{1400} + \frac{0{,}46 \times 0{,}95}{2200}\right) 54 \times 31{,}54 \times 10^6 = 0{,}80$$ million m³.

*Method three*. The amount of tractional load in the reservoir, which is 46% of the total volume of suspended sediment, will be 0.24 million m3. Consequently, in the third method, the total sediment volume deposited in the Aparan reservoir over 54 years will be 0.45 million m³.

## V. DISCUSSION

Thus, according the suggested three methods of calculation three values of the volume of sediment deposited in the reservoir were obtained: according to the first method 0.31 million $m^3$, according to the second - 0.80 million $m^3$ and according to the third - 0.45 million $m^3$, respectively,. The smallest value corresponds to the calculations carried out on the basis of the measured values of the suspended sediment flow of the Kasakh River. The other two volumes of sediment are derived from hydrological calculations. Logically, the first value should be more accurate. However, the hydrological record of measured flows (Table 1) compared to the fluid flow of the river (Table 2) is very short, which does not allow the result of the first method to be taken as a basis and consider it as a most correct one.. Comparison of the calculation results with actual data will allow assessing the degree of reliability of each calculation method and identify the reasons for possible discrepancies.

To carry out field studies on the basis of a topographic map of the area, a 2-D model of the Aparan Reservoir was developed before it was put into operation. The characteristic sections were selected and the areas connecting the model and the terrain were marked. The measurements in the reservoir were carried out during the period when the useful volume of water (81 million $m^3$) was used and the water level in the reservoir dropped to DWL. A rover was used to determine the coordinates of the sediment surface on the dried-up part of the reservoir (Fig. 2), and in the dead volume, a depth gauge equipped with GPS was used (Fig. 3).

Coordinates of 398 points have been measured, 360 of which in the dried-up part, and the remained - in the dead volume. These data were transferred to the produced 2-D model of the reservoir and 2-D and 3-D models of the reservoir bed surface were developed, respectively, before the reservoir was put into operation and at the present time (Figs. 4 and 5).

In Fig. 5 dark color indicates areas of sediment deposition. The upper section of deposits with a length of about 1.1 km is formed by sediment transported by the main stream of the Kasakh River. Their volume is 0.69 million $m^3$. They are entirely located in the usable volume of the reservoir.

The lower section of deposits with a length of about 0.8 km is mainly formed by sediment of the left and right tributaries of the Kasakh River (lower part of Fig. 4).The volume of the deposit is 0.63 million m3. Most of them (0.4 million m3) are located in the dead volume area. Between these areas, over a length of about 0.9 km, there are practically no deposits; the original reservoir bottom remained unchanged.

Thus, the total amount of sediment deposited in the Aparan reservoir is 1.32 million $m^3$. Of this amount, 0.92 million $m^3$ is used for the useful and 0.4 million $m^3$ for the dead volume of the reservoir.

When comparing the calculated and field study results, it is necessary to take into account that the calculated three values of the sediment volume were obtained on the basis of hydrological and hydrometric data of the Kasakh River. The influence of the sediment behavior of the two tributaries of the river on the reservoir was not taken into account due to the lack of any measured data on these waterways. Based on this, the values of the calculated volume will be compared with the actual volume of the upper section of the sediment obtained from field studies, i.e. with a value of 0.69 million $m^3$.

*Fig. 2:* Coordinate measurement of the deposit surface by the rover

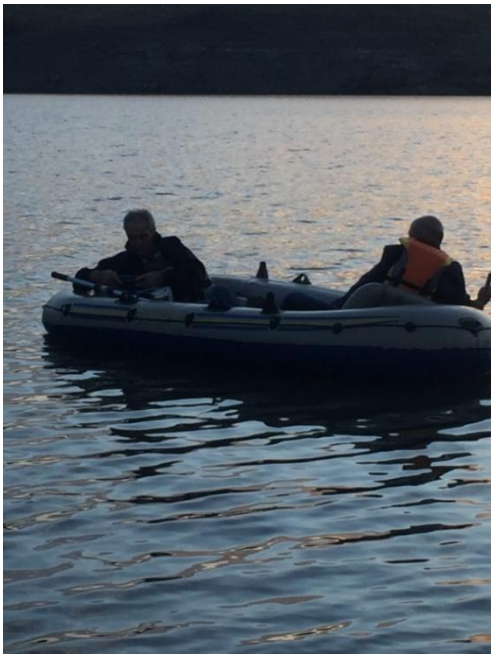

*Fig.3:* Measuring coordinates of the deposit surface with a deep gauge

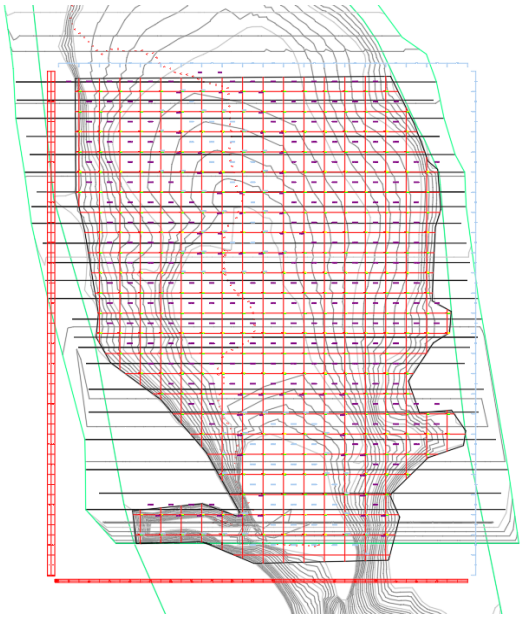

*Fig.4. 2:* D model of the reservoir bowl

*Fig.5. 3:* D model of the reservoir

According to the above described three calculation methods the following volumes were obtained, respectively: 0.31 million m³; 0.45 million m³ and 0.80 million m³. The first two calculated values of the volume of deposits are significantly smaller than the actual one. Such a discrepancy in the case of the Kasakh River can take place: as a result of a reduced estimate of the volume of the sediment settled down to the of the water body bottom (it is assumed to be 46% of the amount of suspended particles); due to inaccuracies in the course of measuring the flow of suspended sediment and in the mapping of the turbidity of regional waterways; and finally, in the presence of the above both disadvantages.

As for 0.80 million m³ of the volume calculated according to Eqs. (1) and (2), its difference with the actual volume of 0.69 million m³ is 14%. The resulting small discrepancy shows that under the

conditions of the problem under consideration, the third method gives fairly good results. For the final assessment of the calculation methods applicability, it is necessary to carry out similar studies in a number of other reservoirs in the region.

The proposed methods of cleaning and repairing can be used both for operating reservoirs and those planned for the future. Based on the forecast of the characteristics of their siltation, it is very important to carry out measures for its cleaning. Analysis of the results of studies of the sediment regime of a number of rivers and the process of siltation of reservoirs in the Republics of Armenia and Artsakh allowed the authors to propose a number of engineering measures that significantly slow down the process of sediment deposition and reduce the useful volume. Among them:

*Engineering measures*

1. In the case of the construction of a new reservoir or for a slightly silted existing reservoir, the amount of sediment carried along by the flow can be significantly reduced with the help of a sediment-retention waterworks facility, which can be installed above the reservoir or in the riverbed itself or next to it. Bottom sediments and large suspended particles will be deposited in its pre-calculated volume. The accumulated sand and gravel soil can be periodically removed and used for construction. A similar solution was used in the design of a small reservoir on the river Selav Mastara (RA).
2. If the reservoir is sufficiently silted up, then in order to improve its regulated capacity, it is necessary to plan measures for the partial or complete cleaning of sediment deposits, which can be carried out mechanically or by hydraulic flushing. The first method is quite laborious and expensive, so mechanical cleaning can be used for small reservoirs.

To clean relatively large reservoirs, it is advisable to use the hydraulic flush method. In the practice of operating these structures, this method has been used. For example, the reservoir Matagis, built on the river. Tartar (Republic of Artsakh). For 40 years of operation, more than 60% of its volume was occupied by nanosam. Due to the accident of the bottom outlet gate, the river runoff in a dense layer of sediments washed away and carried away more than 500 thousand m3 of soil for two years (Fig. 6, 7). The width of the flushing ravine varied within 70-120 m, the average depth was 9-10 m.

In both measures to clean up sediment deposits, to improve the filtration protection of the reservoir bottom, its surface can be covered with a waterproofing layer of the mixture created using pump materials and polymer-mineral material (PMM). Such a layer of 10-15 cm is easy to fit and does not require a lot of resources.

*Fig. 6:* Flushing ravine in the sediments of the Mataghis reservoir

## VI. CONCLUSIONS

Comparison of the calculated and measured values of the sediment volume with the design values of the total and useful volumes of the reservoir (respectively, 91 million $m^3$ and 81 million $m^3$) shows that the total volume of sediment in any case does not exceed 1.5% of the total volume of the reservoir. It means that during 54 years of operation, the design parameters of the Aparan Reservoir underwent very small changes. The result is unexpected, since studies in a number of reservoirs of the mountain-foothill zone show [Baljyan P.H. et al. (June, 2014), Baljyan P.H. & Kelejyan H.G. & Poghosyan A.A. & Namatyan N.T. (2020)] that during the same period of sediment deposition in them occupies a significant part of the basin of the reservoir (10 to 20%). And in some cases, this percentage reaches up 50 to 60%.

The third method is more reliable for assessing the sediment regime of the river under the conditions of the Aparan Reservoir. During 54 years of operation, sediment occupied 1.5% of the total volume of the reservoir. At the same time, the useful volume decreased by 1.1%, and the dead volume - by 4.0%. It can be stated that the volumetric characteristics of the Aparan Reservoir have undergone little changes during the operation period. The results obtained show that the alluvial regime of the Kasakh River is quite weak and very favorable for structures, accumulating river runoff. It can be assumed that in the course of further operation, the regulatory characteristics of this structure will not undergo marked changes.

The methods proposed in the article can also be used for other reservoirs, and allow, based on the forecast of their siltation, to plan and implement measures for their cleaning or making other management decisions. In particular, it is possible to clean up part of the islands created due to sediment in the summer, and thereby increase the useful volume of the reservoir, which can be used for irrigation of agricultural land. For reliability, the cleaned bottom of the reservoir can be covered with a waterproofing layer of the mixture created using pump materials and polymer-mineral material (PMM) [SarukhanyanA.A. & Vartanyan A.A., & Veranyan G.G. & Tokmajyan H.V. (November, 2020), Tokmajyan Vache H. & Vardanyan Arevshad A. & Galstyan Armavir G. & Miqayelyan Nver A. (2020)]. Such a tamped layer of 10-15 cm is easy to fit and does not require large expenditures of resources.

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# Investigation of Clay Deposits as Supplementary Cementitious Materials (Scms): Case Study of Songololo Deposit, Kongo central/d.r. Congo

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## ABSTRACT

Nowadays, the Cement industry is relying on Supplementary Cementitious Materials (SCMs) to reduce the clinker factor, enabling the reduction of carbon dioxide ($CO_2$) emission into the atmosphere, but this will be a safer solution only if it's using available and abundant material as SCMs. The discovery of alternatives to conventional SCMs is a big challenge for sustainability.

The thick clay deposits (namely YC, RC, and LC) aged Neoproterozoic of the West-Congolian group and widely spread in the Songololo area (DR Congo), where five cement plants are erected, are examined in this paper to produce the calcined clay (metakaolin) which can be then mixed with limestone to partially substitute to the clinker in the cementitious system and make an eco-cement called Limestone Calcined Clay Cement (LC3).

The initial clay assessment based on chemical composition was carried out using the XRF method, then the phase composition using the XRD method, and finally, the Kaolinite Equivalent (KEQ) parameter to determine the kaolinite content of each clay using a laboratory oven.

*Keywords:* clay, calcined clay, limestone, lc3 cement, songololo.

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# Investigation of Clay Deposits as Supplementary Cementitious Materials (Scms): Case Study of Songololo Deposit, Kongo central/d.r. Congo

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## ABSTRACT

*Nowadays, the Cement industry is relying on Supplementary Cementitious Materials (SCMs) to reduce the clinker factor, enabling the reduction of carbon dioxide ($CO_2$) emission into the atmosphere, but this will be a safer solution only if it's using available and abundant material as SCMs. The discovery of alternatives to conventional SCMs is a big challenge for sustainability.*

*The thick clay deposits (namely YC, RC, and LC) aged Neoproterozoic of the West-Congolian group and widely spread in the Songololo area (DR Congo), where five cement plants are erected, are examined in this paper to produce the calcined clay (metakaolin) which can be then mixed with limestone to partially substitute to the clinker in the cementitious system and make an eco-cement called Limestone Calcined Clay Cement (LC3).*

*The initial clay assessment based on chemical composition was carried out using the XRF method, then the phase composition using the XRD method, and finally, the Kaolinite Equivalent (KEQ) parameter to determine the kaolinite content of each clay using a laboratory oven.*

*The composite cement (LC3) was prepared by mixing calcined clay, limestone, clinker, and gypsum at different clinker substitution rates. Physical analysis of the composite cement was performed, standard mortars were prepared in accordance with the Norm EN 196-1, and finally, strength development was measured at 1, 2, 7, 28, and 90 days to monitor the pozzolanic activity of the calcined clay – limestone mixture.*

*Among three types of clay of Songololo deposit, the results highlight that YC clay is fit, after calcination at 850°C, for use as SCM. Strength development of the mortar prepared using calcined YC clay complies with the standard EN 197-1 and is even better than the reference cement (Cimko 32.5 and Cimko 42.5). Thus, YC clay can be considered immediately for future development of LC3 cement, while further studies are recommended for LC clay. RC clay has low calcination prospects.*

*Keywords:* clay, calcined clay, limestone, lc3 cement, songololo.

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## I. INTRODUCTION

The Songololo deposits aged Neoproterozoic of West-Congolian group is characterized by thick (ca35m) clay deposits overlapping limestone or conglomerate rocks [1]. Scarce information on these clays exists, and there is no industrial valorization at a large scale. A small quantity is consumed either

by the local cement industry (ca3% of the removed quantity) or by villagers in fired clay bricks production, in housing, and in road stabilization. Still, the significant quantity is rejected by the cement producers and dumped as waste in open fields leading to land and water pollution, and landscape modification.

The cement industry requires only tiny quantity of silica-rich clay to produce ordinary Portland cement (OPC), which cement is primarily consumed worldwide. Cement is a cheap material, easy to handle, and available, but, its production is responsible for high $CO_2$ emissions (5 to 8% of global anthropogenic emissions, and around 35% of industrial emissions). On the average, 0.8 to 0.9 tons of $CO_2$ are emitted to produce one ton of OPC [2].

Many techniques have been developed in order to tackle this environmental issue, but in this paper the partial substitution of clinker with the calcined clay-limestone mixture is emphasized by investigating Songololo clay deposits and their potential utilization as SCM in LC3 composite cement.

The Figure 1 shows the study location.

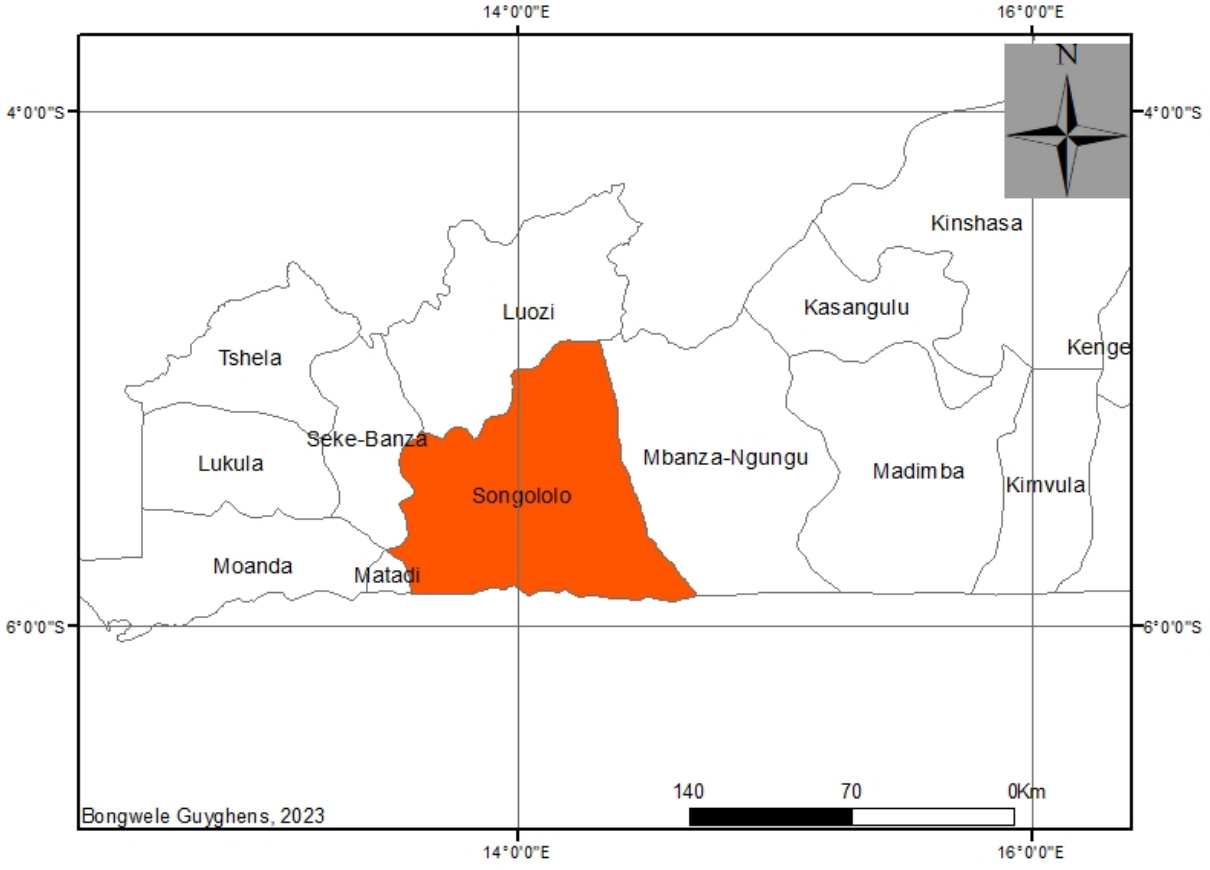


*Figure 1:* Kongo central Province, DR Congo showing study location [1]

### *1.1 Clay Minerals*

Clay minerals are sheet minerals composed of repetitive tetrahedral silicate (T) and octahedral aluminate (O) layers. They are often referred to in the literature as 1:1 (T-O) and 2:1 (T-O-T) minerals.

Minerals 1:1 comprise two groups: (i) the kaolin group, which includes kaolinite, dickite, nacrite, halloysite, and hisingerite, while (ii) the second group includes, among others, lizardite, antigorite, chrysotile, caryopilite, pyrosmalite, and serpentine [3].

Minerals 2:1 form a broad group that includes pyrophyllite, talc, micas, illites, smectites (montmorillonite), vermiculite, chlorites and minerals with a mixed layered structure [4, 5, 6, 7].

This division is far from covering the rich diversity of clay minerals, as clays also contain complex minerals in which T-O and T-O-T layers coexist and can be randomly distributed [8, 9]. To sum up, in the cement industry, three main types of clay are described: (i) kaolinite; (ii) Illite; and (iii) smectites (montmorillonites).

Kaolinite differs from other types of clays, such as illite or smectite, by its layer structure. Kaolinite is a 1:1 clay which is make of one octahedral sheet and one tetrahedral sheet, while illite and smectite are 2:1 clay. Hydrogen bonds between the tetrahedral silicate and the octahedral aluminate prevent any water to penetrate the interlayer space, characterizing kaolinite as non-swelling clay. [10, 11].

### *1.2 Activation of Clays*

Natural clays usually have a moderate or low pozzolanic activity. To increase it, they need to be activated either by grinding [13, 14] or by heating to a temperature which can destroy the crystalline structure and create disorder, and at the same time which can avoid the recrystallization and the formation of chemically inert phases [11, 17].

The clay minerals which can be easily heat-activated are kaolinite and montmorillonite [8, 10]. During heat activation, clay minerals undergo processes of dehydroxylation and amorphisation, leading to a serious damage in the crystal structure. These bring changes in coordination of Al and Si ions, and increase both their solubility and reactivity, which is an essential condition for pozzolanic activity of clay minerals [11].

This effect is more pronounced in the case of kaolinite than in the case of group 2:1 minerals. The higher pozzolanic activity of calcined kaolinite results from the position of Al atoms after dehydroxylation process which remain exposed on the surface of the crystal structure and enable easy reaction with cement hydration products [12].

But, this is not the case for Illite and montmorillonite in which Al atoms remain inside the crystal structure after dehydroxylation process, and this don't facilitate the contact with cementitious materials during cement hydration [12]. As result, their pozzolanic activity is lower than that of calcined kaolinite [10].

Additionnally, muscovite, which belongs to the same family as illite, can be heat-activated, but unfortunately develops low pozzolanic activity compared to illite.

## II. EXPERIMENTAL SET UP

The raw materials needed to produce Portland LC3 composite cement are OPC clinker, limestone, kaolinite clay, and gypsum. In this paper, the natural clay is emphasized. Macroscopically, there are three types of clay in the Songololo deposits, namely YC, RC, and LC, discriminated by their color and texture, as shown in the pictures below. The clay samples were collected and dried before being ground for activation. The clay powders were analyzed for major oxide content using a Thermo Scientific ARL 9900 series IntelloPower X-ray fluorescence spectrometer (XRF).

The different phases (mineral and amorphous) present in the clays were analyzed with a Philips PW1050 diffractometer using graphite-monochromatized Cu-Kα radiation (λ = 1.5418 Å) in the 2θ 4-65° range (step length: 0.02° 2θ, scan time: 5s; 40 kV and 20 mA) and the Rietveld method was used for quantification of the different phases.

The kaolinite equivalent parameter (KEQ) of the different clays was determined using a small laboratory oven for the kaolinite content of each clay sample. 500g of each clay was calcined at 200°C, 350°C and 850°C respectively. The residence time of the clays at these temperatures was 5 min. The optimum calcination temperature for the activation of the natural clays was adopted from Onanga et al, 2023 [1].

The composite cement was prepared by mixing calcined clay, clinker (OPC), limestone powder, and gypsum, gradually reducing the proportion of clinker in the mix (50 - 65%). Automatic mixer (1600rpm) was used to obtain a homogeneous cement.

The mortars were prepared under EN 196-1 standard, and the pozzolanic activity of the calcined clay-limestone mixture was analyzed using the indirect method by measuring the compressive strength developed by the mortars on days 1, 2, 7, 28, and 90. Different compressive strengths were compared with those developed by reference cement (Cimko 32.5 and Cimko 42.5) and then with EN 197-1 Norm. This helps to select the suitable clay for developing LC3 cement.

### *2.1 Tests of Eligibility of Clays for Activation Procedure*

Clay is a material that is widespread throughout the world, inexpensive, and easily accessible [8]. Clays are very diverse in terms of chemical and mineralogical compositions making clay with only one type of clay mineral very rare, as [9, 11]. Thus, it is essential to analyze the different clays chemically and mineralogically in order to select which comply with LC3 requirements [18].

Diaz et al. studied clays for their use as SCMs for LC3 production and established a series of chemical criteria to select the suitable clay. Ttable 1 shows the chemical criteria for the suitability of clays in the LC3 system [72].

*Table 1:* Chemical acceptance of Clay for LC3 system

| | $Al_2O_3$ | $Al_2O_3/SiO_2$ | LOI | CaO | $SO_3$ |
|---|---|---|---|---|---|
| Suitable Cay | > 18% | > 0.3 | < 7% | < 3% | < 3% |

However, chemical composition itself is not enough to explain the pozzolanic activity of clays. To take into account the later, the parameter defined as kaolinite equivalent (KEQ) was determined and calculated according to Equation: [18]:

$$\%KEQ = \{[m(350\ °C) - m(850\ °C)]/[m(200\ °C) \times 0.1396]\} \times 100, \quad \text{(Equation 1)}$$

In which m(x) is the mass of the mineral after heat treatment at a given temperature x. The suitable clay must have at least 40% KEQ.

### *2.2 Methods of Assessment of Pozzolanic Activity*

The pozzolanic reaction occurs, in case of LC3 cement, between the aluminosilicates in the clays ($Al_2O_3$-$2SiO_2$) and the portlandite (Ca (OH) $_2$) produced by cement hydration, and the hydration products are CSH gel ($CaO$-$SiO_2$-$H_2O$), hydrated calcium aluminates (CAH), as well as hydrated calcium aluminosilicates of the hydrogehlenite ($C_2ASH$) and hydrogarnets ($C_3AS_3$-$C_3AH_6$) type. With the addition of calcite, carbo-aluminates can also be formed [19, 20, and 21]. The performance of mortar depends on the different type and the proportion of hydrated products at different ages.

The pozzolanic activity of materials is assessed either by direct or indirect methods. Direct methods are based on the measure of the portlandite ($Ca(OH)_2$) content and its reaction products over the time in the solution [22, 23], while indirect methods analyze the evolution of some specific parameters which

can be, for example, the compressive strength of mortar specimens, the electrical conductivity of a saturated solution of portlandite [$Ca(OH)_2$] in which the material tested is placed [24, 25], or the determination of the amount of heat released in calorimetric tests [84,85]. In this paper, the pozzolanic activity of the calcined clay – limestone mixture is determined by measuring the compressive strength developed by the mortar during days 1, 2, 7, 28 and 90.

## III. RESULTS AND DISCUSSION

### *3.1. Chemical Composition*

Unlike Ordinary Portland Cement (OPC) which requires siliceous clay, the LC3 cement consumes high alumina clay, and this can be observed in the $Al_2O_3$ / $SiO_2$ ratio of the natural clays (See Table 2). YC and RC clays have high $Al_2O_3$ contents and less $SiO_2$, whereas RC clays have very high $SiO_2$ contents. Considering chemical acceptance limits of Diaz et al. (see Table 1) [18], YC and RC clays have good calcination potential and can be considered for future studies.

*Table 2:* Chemical Composition of Different Clays

| Clay Samples | | Chemical Composition (%) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $SiO_2$ | $AI_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $TiO_2$ | $Mn_2O_3$ | $P_2O_5$ | LOI | $Al_2O_3$ / $SiO_2$ |
| YC | 1 | 57,38 | 20,36 | 9,1 | 0,13 | 1,07 | 0,31 | 1,11 | 0,16 | 0,35 | 7,32 | 0,35 |
| | 2 | 57,5 | 20,34 | 9,8 | 0,15 | 1,05 | 0,29 | 1,11 | 0,17 | 0,4 | 7,4 | 0,35 |
| | 3 | 56,9 | 20,2 | 9,6 | 0,13 | 1,06 | 0,3 | 1,09 | 0,15 | 0,37 | 7,35 | 0,36 |
| | 4 | 57,5 | 20,12 | 9,7 | 0,14 | 1,04 | 0,28 | 1,12 | 0,16 | 0,28 | 7,34 | 0,35 |
| RC | 1 | 75,03 | 12,6 | 5,98 | 0,01 | 0,21 | 0,4 | 0,68 | 0,04 | 0,14 | 5,17 | 0,17 |
| | 2 | 76,2 | 12,8 | 5,04 | 0,01 | 0,19 | 0,39 | 0,59 | 0,05 | 0,15 | 5,05 | 0,17 |
| LC | 1 | 33,91 | 17,07 | 37,08 | 0,11 | 0,21 | 0,31 | 0,77 | 0,06 | 0,31 | 10,48 | 0,50 |
| | 2 | 33,95 | 18,01 | 36,06 | 0,12 | 0,2 | 0,3 | 0,76 | 0,05 | 0,35 | 10,5 | 0,53 |

The other components used in the LC3 cement system have also been analyzed, and Table 3 gives the chemical composition of these components.

*Table 3:* Chemical composition of different components

| Component | Chemical Composition (%) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $AI_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $K_2O$ | $Na_2O$ | LOI |
| Limestone | 4,50 | 0,89 | 0,50 | 50,08 | 2,06 | 0,34 | 0,19 | 0,12 | 42,68 |
| Gypsum | 2,28 | 0,63 | 0,42 | 31,01 | 1,93 | 40,63 | 0,20 | 0,07 | 22,85 |
| Clinker | 21,04 | 5,05 | 3,52 | 65,17 | 1,80 | 0,93 | 0,76 | 0,20 | 0,31 |

### *3.2 Phase Content*

The YC clay has the highest kaolinite content (36% on average), followed by the LC clay (30% on average) and, the RC clay has the lowest (25% on average). phase content.

Nevertheless, the clay mineral content (kaolinite, illite, and muscovite) exceeds 50% in the YC clay and is only 33% and 27%, respectively, in the LC and RC clays. Also, the amorphous phase represents 9% in YC clay, 14% in RC clay, and is not present in RC clay.

Quartz is found in all clays, and its influence on mortar performance remains to be determined.

Table 4 highlights the XRD result of different clay, and Table 5 shows the Kaolinite Equivalent (KEQ) of each clay and the mass loss after calcination.

The mass loss after calcination at 850 °C varies for all clays from 11.2 to 11.5%, and this highlights the clay mineral diversity in the Songololo deposit (See Table 6).

*Table 4:* Mineralogical Composition YC Clay

| Phase (%) | Formula | Clay Samples | | | | | |
|---|---|---|---|---|---|---|---|
| | | YC1 | YC2 | RC1 | RC2 | LC1 | LC2 |
| Quartz | $SiO_2$ | 31,6 | 30,1 | 68 | 67,3 | 20,4 | 20,1 |
| Muscovite | $KAl_2AlSi_3O_8(OH)_2$ | 3,3 | 2,9 | 2 | 2,3 | 3,1 | 3 |
| Kaolinite | $Al_2Si_2O_5(OH)_4$ | 36,4 | 37,5 | 25,4 | 26,2 | 31,6 | 30,5 |
| Illite | $(K.H_3O))Al.Mg.Fe)_2(Si.Al)_4O_{10}[(OH)_2.(H_2O)]$ | 16,2 | 15,3 | - | - | - | - |
| Goethite | FeOOH | 4,3 | 4,2 | 3,3 | 4,2 | 27,8 | 28,7 |
| Hematite | $Fe_2O_3$ | 0,4 | 0,3 | - | - | 3,2 | 3,1 |
| Amorphous | | 9,6 | 9,7 | - | - | 13,9 | 14,6 |

*Table 5:* KEQ of Different Clays

| Sample | Initial mass (g) | Mass(g) @ 200 °C | Mass (g) @ 350°C | Mass(g) @ 850°C | K | KEQ (%) |
|---|---|---|---|---|---|---|
| YC 2 | 500 | 475 | 467,5 | 442,5 | 0,1396 | 37,70 |
| YC 3 | 500 | 475 | 467 | 442,5 | 0,1396 | 36,95 |
| YC 1 | 500 | 475 | 466 | 442,5 | 0,1396 | 35,44 |
| | | | | | | |
| RC 1 | 500 | 476 | 460 | 443 | 0,1396 | 25,58 |
| RC 2 | 500 | 475,5 | 460,5 | 443 | 0,1396 | 26,36 |
| RC 3 | 500 | 476 | 461,5 | 443 | 0,1396 | 27,84 |
| | | | | | | |
| LC 3 | 500 | 476 | 464 | 443,5 | 0,1396 | 30,85 |
| LC 2 | 500 | 477,5 | 464 | 444 | 0,1396 | 30,00 |
| LC 1 | 500 | 475,5 | 464 | 443 | 0,1396 | 31,64 |

*Table 6:* Mass lost after calcination of Clays

| | Mass Loss (%) | | | |
|---|---|---|---|---|
| | Initial mass loss (%) | Mass loss (%) @ 200 °C | Mass loss (%) @ 350°C | Mass loss (%) @ 850°C |
| YC 2 | 0 | 5.0 | 6.5 | 11.5 |
| YC 3 | 0 | 5.0 | 6.6 | 11.5 |
| YC 1 | 0 | 5.0 | 6.8 | 11.5 |
| | | | | |
| RC 1 | 0 | 4.8 | 8.0 | 11.4 |
| RC 2 | 0 | 4.9 | 7.9 | 11.4 |
| RC 3 | 0 | 4.8 | 7.7 | 11.4 |
| | | | | |
| LC 3 | 0 | 4.8 | 7.2 | 11.3 |
| LC 2 | 0 | 4.5 | 7.2 | 11.2 |
| LC 1 | 0 | 4.9 | 7.2 | 11.4 |

Considering various recommendations in the literature concerning clays, YC clay offers good prospects for use as SCM in the LC3 system. More testing is required for LC clay, while RC clay has poor prospects.

### *3.3 Pozzolanic Activity*

The pozzolanic activity of the mixture (calcined clay – limestone) in the cementitious system was determined by monitoring the compressive strength developed by the mortars during days 1, 2, 7, 28, and 90. Clinker used came from the Cimko cement plant, limestone from the quarry of the same company, and gypsum was imported from Angola. Table 3 shows the chemical composition of these materials and Table 7 gives the proportion of each material in the formulation of the composite cement.

*Table 7:* Proportion (%) of components in the cement formulation

| | Proportion (%) | | | |
|---|---|---|---|---|
| | Clinker | Calcined Clay | Limestone | Gypsum |
| Cimko 32.5 | 65 | 0 | 32 | 3 |
| Cimko 42.5 | 89 | 0 | 8 | 3 |

| | Proportion (%) | | | |
|---|---|---|---|---|
| | Clinker | Calcined RC | Limestone | Gypsum |
| Trial 1 with RC | 50 | 30 | 15 | 5 |
| Trial 2 with RC | 55 | 25 | 15 | 5 |

| | Proportion (%) | | | |
|---|---|---|---|---|
| | Clinker | Calcined LC | Limestone | Gypsum |
| Trial 1 with LC | 50 | 30 | 15 | 5 |
| Trial 2 with LC | 60 | 22,5 | 12,5 | 5 |

| | Proportion (%) | | | |
|---|---|---|---|---|
| | Clinker | Calcined YC | Limestone | Gypsum |
| Trial 1 with YC | 55 | 30 | 15 | 5 |
| Trial 2 with YC | 60 | 22,5 | 12,5 | 5 |
| Trial 3 with YC | 65 | 20 | 10 | 5 |
| Trial 4 with YC | 65 | 15 | 15 | 5 |

Figures 2-4 show the compressive strength of the mortars prepared with calcined YC, RC, and LC clays. These results are compared both to the reference cement (Cimko 32.5 and Cimko 42.5), and to EN 197-1 standards (Figure 5).Table 8 shows the physical performance of each formulation, and this is also compare to the reference cement. The results show that, the composite LC3 mortar under investigation complies with EN 197-1, and can also compete with the reference cement.

*Table 8:* Physical Analysis of Different Cements

| | Consistence % | Setting time (Min) | Blaine (cm2/g) | Residue (%) | | Soundness (%) |
|---|---|---|---|---|---|---|
| | | | | 45µ | 90µ | |
| Cimko 32.5 | 31 | 190 | 5885 | 18,1 | 0,1 | 1,05 |
| Cimko 42.5 | 30 | 180 | 5829 | 21 | 0,1 | 1,1 |
| Trial 1 with RC | 31 | 190 | 5885 | 18,1 | 0,1 | 1,5 |

| | | | | | | |
|---|---|---|---|---|---|---|
| Trial 2 with RC | 30 | 180 | 5829 | 21 | 0,1 | 1,5 |
| | | | | | | |
| | Consistence % | Setting time (Min) | Blaine (cm2/g) | Residue (%) | | Soundness (%) |
| | | | | 45µ | 90µ | |
| Cimko 32.5 | 31 | 190 | 5885 | 18,1 | 0,1 | 1,05 |
| Cimko 42.5 | 30 | 180 | 5829 | 21 | 0,1 | 1,1 |
| Trial 1 with LC | 30 | 180 | 5500 | 16,2 | 0,1 | 1,5 |
| Trial 2 with LC | 31 | 175 | 5462 | 12,5 | 0,1 | 1,4 |
| | | | | | | |
| | Consistence % | Setting time (Min) | Blaine (cm2/g) | Residue (%) | | Soundness (%) |
| | | | | 45µ | 90µ | |
| Cimko 32.5 | 31 | 190 | 5885 | 18,1 | 0,1 | 1,05 |
| Cimko 42.5 | 30 | 180 | 5829 | 21 | 0,1 | 1,1 |
| Trial 1 with YC | 32 | 190 | 6019 | 14,3 | 0,1 | 1,4 |
| Trial 2 with YC | 32 | 180 | 6395 | 16,7 | 0,1 | 1,34 |
| Trial 3 with YC | 32 | 170 | 5713 | 16 | 0,1 | 1,33 |
| Trial 4 with YC | 32 | 170 | 5084 | 14,5 | 0,1 | 1,35 |

All physical performances of LC3 cement at different clinker substitution rates comply with EN 197-1 and are almost similar to reference cement, except for Normal Consistency (NC), which is slightly higher due to adding clay.

The initial setting time ranging from 170 to 190 minutes is close to reference cement. The Blaine fineness is higher than reference cement, which may increase compressive strength of the mortar at late age, and the soundness is within the acceptable range even at 90 days.

Please note that no chemical additives have been added to improve mortar performance.

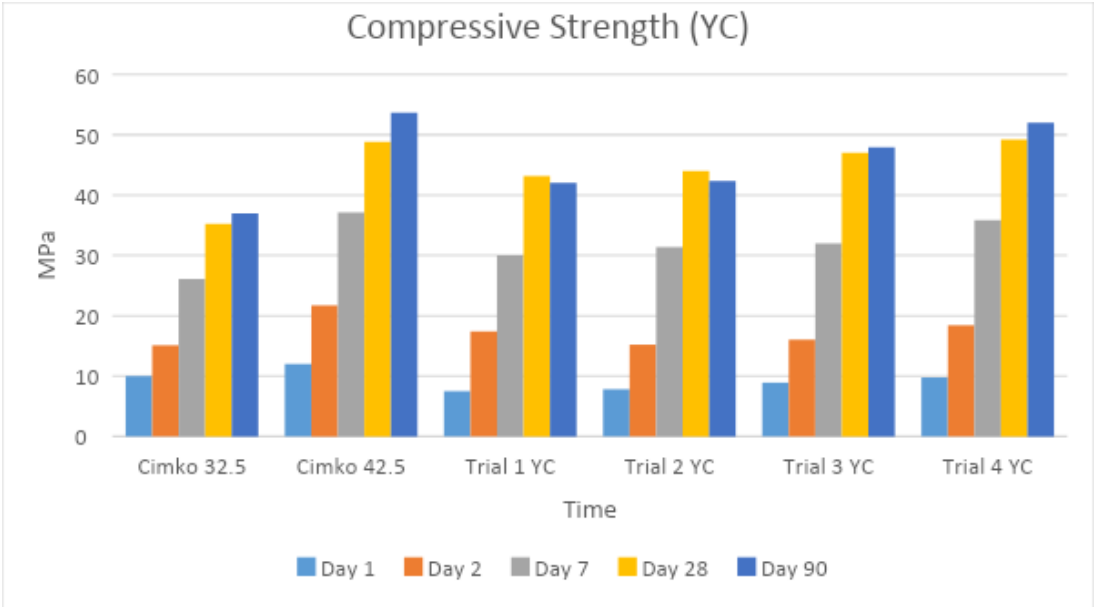


*Figure 2:* Compressive strength of the mortar designed using YC Clay

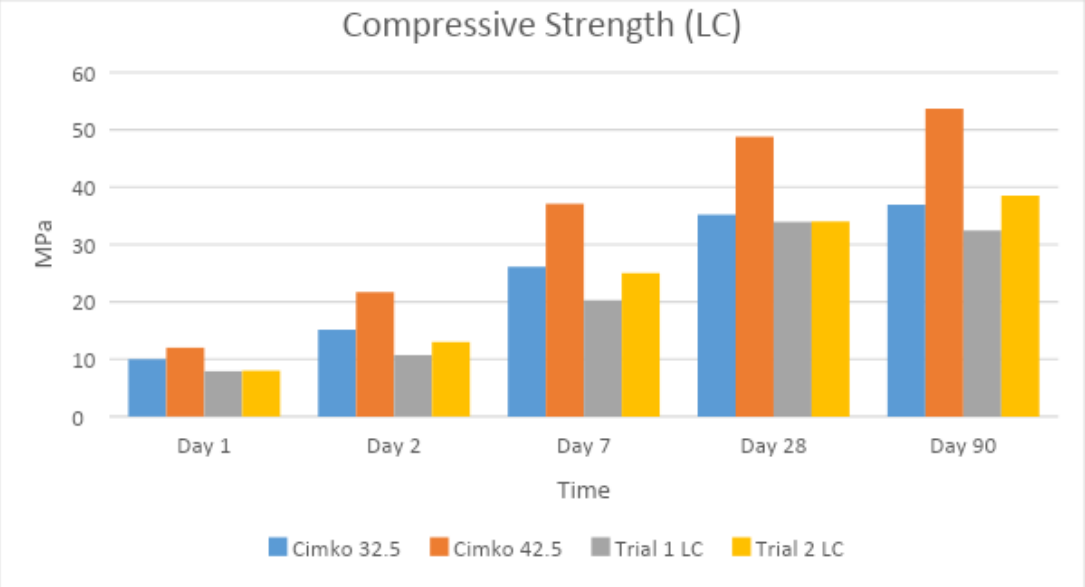


*Figure 3:* Compressive strength of the mortar designed using LC Clay

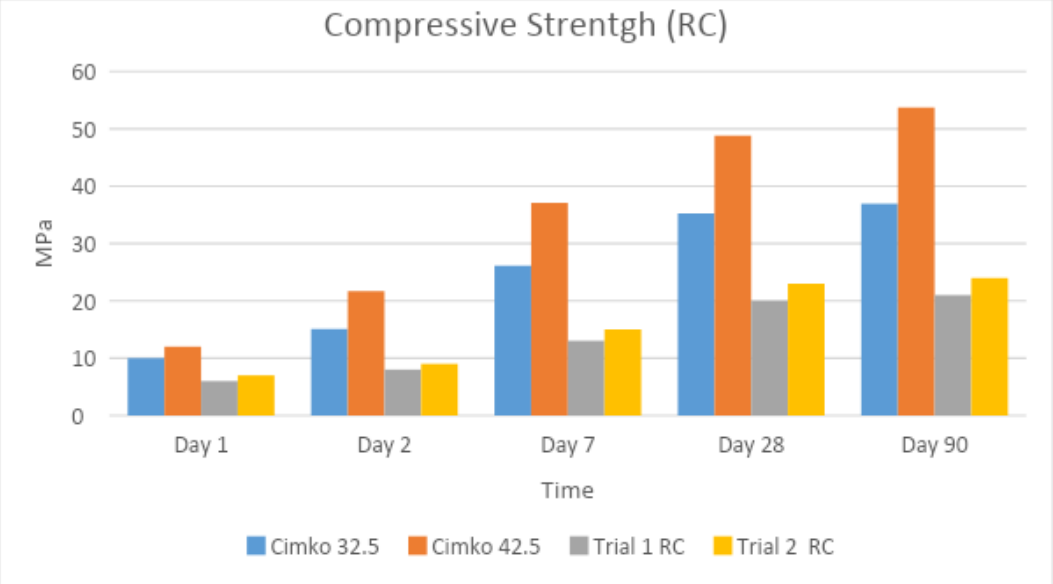


*Figure 4:* Compressive strength of the mortar designed using RC Clay

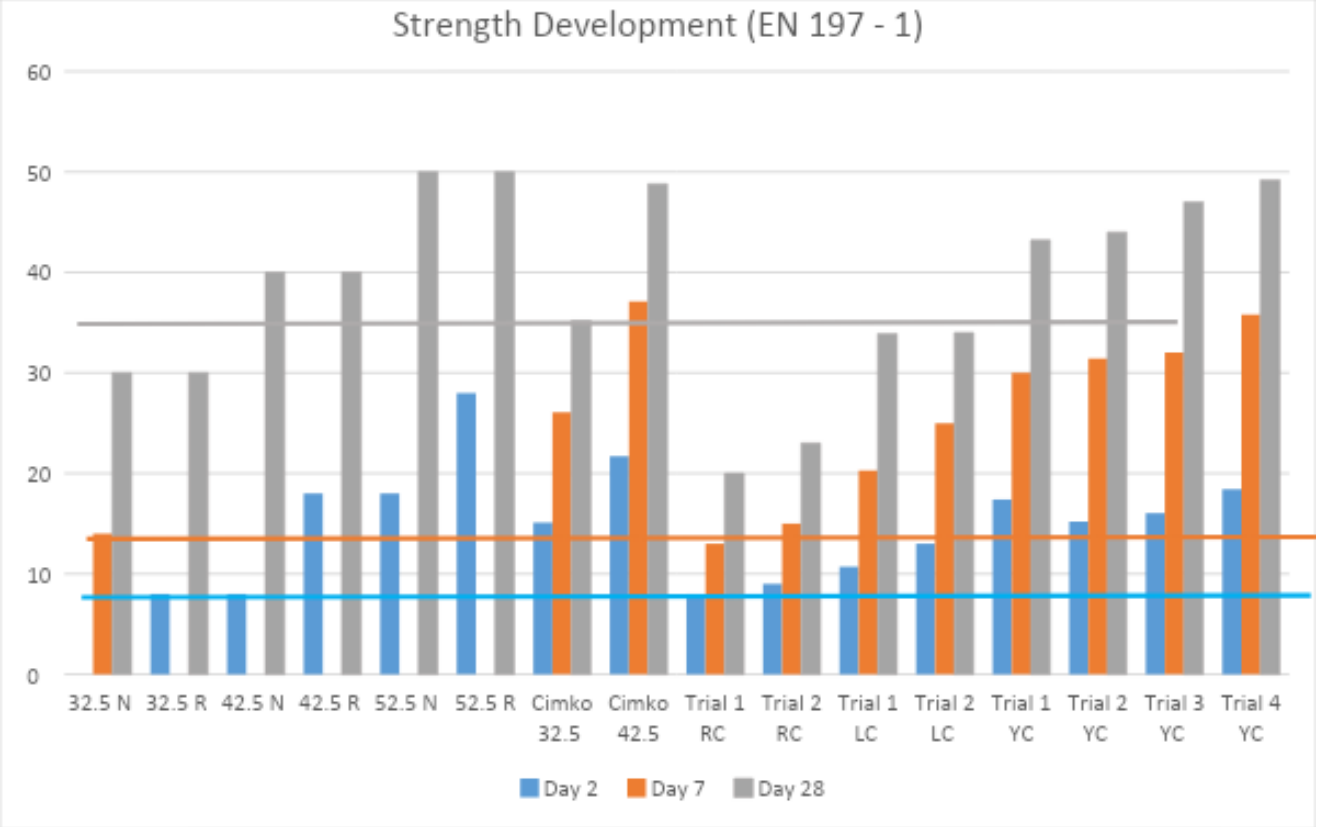


*Figure 5:* Compressive strength as per Norm EN 197-1 [29]

The performance of mortars prepared with calcined RC clay did not meet the requirements of EN 197-1, while mortars based on calcined LC and YC clay achieved performance in line with the requirements of the said standard.

Mortars prepared with calcined LC clay can compete with Cimko 32.5 but do not achieve the performance of Cimko 42.5, while mortars based on calcined YC clay far exceed Cimko 32.5 and can compete with Cimko 42.5.

## IV. CONCLUSION

Three clay types labelled YC, RC, and LC of Songololo deposit were tested for their potential use as SCM in the LC3 cement system after calcination.

Eight clay samples were analyzed for their chemical composition using the XRF technique, the phase content (mineral and amorphous) of the natural clay were determined by XRD, and finally, the Kaolinite equivalent (KEQ) of clays determined by laboratory oven at different calcination temperatures. The composite cement (LC3) was prepared by mixing calcined clay, limestone, clinker, and gypsum at various clinker substitution rates. Reactivity of the calcined clay – limestone mixture was investigated by measuring the compressive strength developed by the mortar after 1, 2, 7, 28, and 90 days.

The results highlight that Songololo deposits are characterized by low kaolinite clays (less than 40%), but among them YC clay has the highest kaolinite (ca 37%), co-occurring with illite and smaller amount of muscovite. Rc clay consisted of low kaolinite content (about 25%) and high quartz content, and finally, the LC clay consisted of moderate kaolinite content and high iron content.

The study of pozzolanic activity of calcined clay – limestone mixture allowed us to formulate the conclusion that with clinker substitution rate (between 40 and 45%), the cementitious material with satisfactory parameters can be obtained and this offers an excellent prospect for industrial trials.

Of the three Songololo deposit clays, only the YC clay can be immediately taken into account in the formulation of LC3 cement. Further studies are recommended for LC clay, while RC clay have limited calcination potential for use as SCMs.

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