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Formulation and preliminary characterization of a biopolymer-based matrix from *Moringa Oleifera* seed powder

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ABSTRACT

Environmental benefits of biopolymers are attractive features in matrix or composite formulation and therefore well sought after in recent times. A variety of sources based on agricultural plant or animal products have been identified and utilized in numerous areas such as drug delivery systems, adsorption, biofiltration water purification etc. Their incorporation in biodegradable matrix appeared favorable since this would result in a completely biodegradable composite. In this work, matrix of Moringa Oleifera Seed Powder (MOSP) and an iron salt, Ferrous Sulfate Heptahydrate (FSH) was formulated and characterized for improved properties and peculiar applications. Chemical, physical and other functional parameters such as swelling, solubility and pH were investigated and excellent properties were exhibited. The synergistic blend effect of MOSP with the highly acidic Iron salt formulated in this work would enhance performance capacity in waste water stabilization, inhibit the growth of odor causing bacteria in animal farms and the environment in general, and hence serve as cheap and effective pollution control agent.

Key words: *Moringa oleifera*, biopolymer, formulation, matrix, characterization.

INTRODUCTION

Biopolymers are renewable and attractive materials due to their availability, biocompatibility, and biodegradation mechanisms [1]. A variety of sources based on agricultural plant or animal products have been identified and utilized in numerous areas. For example, the use of biopolymers as adsorbents for the removal of heavy metals and organic pollutants from water is widely reported [2; 3]. Their major limitation is the fact that they degrade or denature soon after processing, storage and application. It is difficult to envision biopolymers in their unaltered or unmodified state having broad industrial relevance. The formation of composites or matrices by incorporating biopolymers into blends with synthetic materials and the use of crosslinking agents are successful options to create durable bio-based materials [1]. In this work, a blend of MOSP and an iron salt was formulated and characterized.

Moringa oleifera is well known for its broad applications and is reported to be non-toxic [4]. It is drought tolerant and has nutritional, medicinal and water cleaning attributes [5]. The leaves, flowers, fruits and roots are used locally as food articles. The seed pods have been employed as natural adsorbents for organic pollutants in waste waters [2]; [6]. The seeds have also been reported to have strong antimicrobial properties [7]; [8]; [9].

Ferrous sulfate hepta – hydrate is a green and odorless crystalline inorganic powder. It is an economic raw material for waste water treatment. Its potential lies in its good flocculation and decolorization ability, removing the heavy metal ions, oils and phosphorus, disinfection due to high acidity and other functions [10]. A blend of MOSP with alum for water purification has been reported [11].

The present research investigates the characteristic profiles and the synergistic blend effect of MOSP with the highly acidic Iron salt formulated in this work. The combined effect would enhance performance capacity in waste water stabilization, inhibit the growth of odor causing bacteria in animal farms and other municipal solid waste in general, and hence serve as cheap and effective pollution control agent.

MATERIALS AND METHODS

Materials

M. Oleifera pods were obtained from a personal farm in Girei Local Govt area of Adamawa state. The pods were deshelled to obtain the seeds which were then sun dried for five days. They were finally ground in a micro hammer mill. Ferrous sulfate heptahydrate was obtained from Northern Scientific Co ltd and used without further purification.

Formulation of the matrix:

The biopolymer based matrix was formulated from a mixture of MOSP and ferrous sulfate heptahydrate (FSH) crystals by physical blending. The materials were first oven dried according to standard methods, ground to fine powder and sieved using 200 mm mesh size. A factorial design approach was used for the formulation of the biopolymer based matrix for characterization. It involved the two components as factors vize: factor A (Ferrous sulfate) at three levels each i.e a_1, a_2, a_3 representing 2%, 5% and 10% and factor B (MOSP) also at three levels each i.e b_1,b_2, b_3 representing 2%, 5%, and 10% (Table 1). This is a 2x3 factorial experiment from which a total of 9 combinations were obtained and were coded F1 – F9.

Table 1: combination of the factors into formulations

Factors	levels		Formulations	
A	a_1 (2%)		a_1b_1	a_1b_1
	a_2 (5%)	a_1	a_1b_2	a_1b_2
	a_3 (10%)		a_1b_3	a_1b_3
			a_2b_1	a_1b_1
		a_2	a_2b_2	a_2b_2
			a_2b_3	a_2b_3
B	b_1 (2%)		a_3b_1	a_3b_1
	b_2 (5%)	a_3	a_3b_2	a_3b_2
	b_3 (10%)		a_3b_3	a_3b_3

Characterization of matrix formulation

Physical characterization

Physical properties considered significant to the performance of the matrix were evaluated. These included sensory properties (Appearance and odor); pH, density, Solubility index and swelling power.

pH

The pH was measured by making a 10% (w/v) flour suspension of each sample in distilled water. Each sample was then mixed thoroughly in a plastic beaker, and the pH was recorded with an electronic pH meter [12].

Density

Densities of the samples were measured according to the method described in [13]. Empty 10 ml capacity measuring cylinders were weighed and filled with the samples. The cylinders were gently tapped on the bench about 40 – 50 times until no further diminution of the sample level. Individual weights and volumes of the cylinders were recorded and sample densities were calculated.

$$\text{Density} = \text{Weight of sample} / \text{Vol. of sample}$$

Swelling power and SOL index

Swelling power (SP) and solubility index (SI) of the formulations were determined in triplicate following the procedure of [14] and [15. Samples (0.1 g) were mixed with 5 ml of distilled water in 10 ml centrifuge tubes, heated for 30min. at 30 and 50^0C while shaking every 5 min. After heating, samples were centrifuged at 3000 rpm for 15 min. Precipitated paste was separated from supernatant and weighed (Wp). Both phases were dried at 105 ^{0}C for 24 h and the dry solids in precipitated paste (Wps) and supernatant (Ws) were calculated. SP is the ratio of the weight of swollen powdered paste after centrifugation (g) to their dry mass (g): $SP = Wp / Wps$

where, Wp = hydrated paste(g) and Wps = dry precipitated paste(g)

The SOL is the percentage of dry mass of solubles in supernatant (Ws) to the dry mass of powdered sample (W0):

$$SOL = Ws / Wo \times 100\%$$

Sensory evaluation [12]

Sensory evaluation was conducted in the Sensory Evaluation Laboratory, Department of Food Science and Technology, Modibbo Adama University of Technology, Yola. A total of 10 panelists, who included students and staff of the Department of Food Science and Technology participated in this study. Evaluations were held immediately samples were formulated. A total of three replications were completed. Formulations were placed in 100 ml white plastic bottles with lids. Panelists were instructed to remove the lid before assessment. The judges evaluated the samples for color and appearance and overall acceptance on a7 point scale ranging from like very much (7) to dislike very much (1), while odor was also judged on a 5 point scale from not offensive (1) to extremely offensive (5).

Chemical characterization of matrix

The formulated biopolymer based matrix was evaluated for chemical properties like carbohydrates (Molisch test), proteins (Ninhydrin test), chlorides (Silver nitrate test), Sulfates (Barium chloride test), and phytochemicals [16]; [17.

RESULTS AND DISCUSSION

Physical and chemical characterization of raw materials

The behavior of a hybrid material is a function of the basic properties of the raw materials. In order to assess the suitability of matrix, one must know the properties of the base materials that formed part of its building blocks. The results of the chemical and physical characterization of the base materials i.e MOSP and FSH are summarized in tables 2 & 3.

Table 2: Physical Characterization of Base Materials

Materials	PH	Density	Colour	Odour	**Solubility Index**				**Swelling Power**			
					H_2O		CH_{30}		H_2O		CH_{30}	
					30^0C	50^0C	30^0C	50^0C	30^0C	50^0C	30^0C	50^0C
Mosp	5.08	0.66	6.00	0.16	13.33^a	36.62^b	33.00^c	3.66^{bc}	3.30^d	4.62^{ed}	3.14^e	1.12^{fe}
FeSo 4.7 H_2O	2.5	-	5.25	0.10	46.42	46.68	-	-	0.00^a	0.00^a	0.00^a	0.00^a

Data – The same raw with different letter differ significantly (P<0.05)

Table 3: Chemical characterization of base materials

S/N	**Name of component**	**MOSP**	**FeSo4.7H₂o**
1	Carbohydrates	++	-
2	Proteins	++	-
3	Chlorides	-	-
4	Sulfates	+	++
5	Saponins	+	-
6	Tannins	-	-
7	Phenols	-	-
8	Flavenoids	-	-
9	Steroids	-	-
10	Glycosides	+	-

The pH, density and Sol of FSH is seen to be higher than that of MOSP, probably because of its inorganic nature. On the other hand, the SP of MOSP is higher than that of FSH. The screening results of both 1^0 and 2^0 metabolites is presented in table 3. The result indicated the presence of saponins (+), alkaloids (+) and glycosides (+). The presence of phytochemicals in MOSP has been widely reported [9]; [8] and [11]. These phytochemicals are responsible for both pharmacological and toxic activities in plants [18]; [19]. However, its applications in local drug therapy, water purification [20] and food fortification [9] have continued to increase with no reports of any adverse effect probably due to low content of the toxic phytochemicals.

Examination of the formulations in the matrix showed similar chemical properties (Table 4) as the base materials. This was expected as the modification was merely physical and thus did not interfere, to a large extent, on the chemical nature of the matrix. However, functional properties and sensitivity to microbial life of the matrices improved greatly which may be due to synergistic effect.

Table 4: Chemical Characterization of Matrix

Matrix	**CHO**	**NH2**	**CL$^-$**	**SO_4^{2-}**	**Alkaloids**	**Tannins**	**Phenols**	**Saponins**	**Flavonoids**	**Steriods**	**Glycosides**
F1	+	+	-	++	+	-	-	+	+	-	+
F2	+	+	-	++	+	-	-	+	+	-	+
F3	+	+	-	++	+	-	-	+	+	-	+
F4	+	+	-	++	+	-	-	+	+	-	+
F5	+	+	-	++	+	-	-	+	+	-	+
F6	+	+	-	++	+	-	-	+	+	-	+
F7	+	+	-	++	+	-	-	+	+	-	+
F8	+	+	-	++	+	-	-	+	+	-	+
F9	+	+	-	++	+	-	-	+	+	-	+

Functional properties of matrix

Table 5 depicts the results of the physical characteristics of the matrix. The pH of F_1-F_9 formulations ranged from 3.51-3.81 and showed no statistical difference (P>0.05). F_7 exhibited the highest pH of 3.81 and F_5 showed the lowest value of 3.51. The bulk density of F_1-F_9 was between 0.70 - 0.86. Again the data are not significantly different (P>0.05). F_3 gave the least value of 0.70 while F1& F8 had the highest ratio of 0.86 respectively. Generally, they were all below $1g/cm^3$.

This is a significant result since density is implicated in packaging and transportation of products.

The formulations exhibited varying Solubility Indices in different solvents at different temperatures (Table 4). In water, SOL at 50^0C were higher (p> 0.05) than values at 30^0C, concurring that SOL increases with temperature. In methanol however, SOL showed no significant difference at the two temperatures and were slightly lower than those in water. The behavior in methanol showed its limited power probably due to low hydrogen bonding. The high SOL values in water may be due to high hydrogen bonding and the presence of the inorganic component of the matrix which varied as the amount is modified in the formulations. The results for swelling power of formulations varied between 6.50 for F_3 to 15.0 F_7 with significant differences at P>0.05 level. The maximum value is about 3 fold increase over that of the MOSP raw material. When carbohydrate biopolymer dispersions are heated, granules' swelling and starch polymers solubilization occur, which influence the functional properties [15]. The solubility of the matrix is in no way a disadvantage, considering the mechanism of action of the individual components.

Table 5: Physical Characterization of Base Matrix

Materials	PH	Density	**Solubility Index** H_2O		CH_3O		**Swelling Power** H_2O		CH_3O	
			30^0C	50^0C	30^0C	50^0C	30^0C	50^0C	30^0C	50^0C
F_1	3.58	0.81	46.67 ±5.77	66.67 ±5.77	66.67 ±5.77	63.33 ±5.77	8.23 ±2.44	12.28 ±2.01	6.72 ±1.80	4.20 ±0.69
F_2	3.54	0.77	30.00 ±10.00	66.67 ±5.77	53.33 ±5.77	46.67 ±5.77	7.11 ±0.63	5.66 ±0.94	3.27 ±0.47	2.92 ±0.17
F_3	3.55	0.70	33.33 ±5.77	43.33 ±5.77	33.33 ±5.77	33.33 ±5.77	9.2 ±1.01	7.05 ±0.83	3.12 ±0.35	2.86 ±0.24
F_4	3.62	0.55	43.33 ±5.77	60.00 ±0.00	63.33 ±5.77	63.33 ±5.77	7.23 ±1.70	8.55 ±2.14	5.28 ±1.05	4.16 ±0.72
F_5	3.51	0.78	56.67 ±5.77	56.67 ±5.77	63.33 ±5.77	56.67 ±5.77	6.25 ±1.40	6.87 ± 1.73	3.14 ±0.55	4.13 ±0.64
F_6	3.42	0.72	16.66 ±5.77	56.67 ±5.77	46.67 ±5.77	36.67 ±5.77	12.11 ±3.37	6.85 ±1.37	3.14 ±0.14	4.21 ±0.32
F_7	3.81	0.84	46.66 ±5.77	70.00 ±10.00	76.67 ±5.77	76.67 ±5.77	18.33 ±0.57	16.33± 0.57	4.50 ±0.86	5.88 ±1.06
F_8	3.58	0.76	26.67 ±5.77	60.00 ±10.00	70.00 ±10.00	63.33 ±5.77	12.17 ±5.92	10.72 ±2.66	3.69 ±0.56	4.97 ±0.61
F_9	3.51	0.86	26.67 ±5.77	63.33 ±5.77	60.00 ±10.00	53.33 ±5.77	11.33 ±9.81	10.66 ±2.36	3.08 ±0.38	4.40 ±0.52

The blending of MOSP and FSH showed characteristic synergies on the functional properties of the hybrid material. Generally, they were seen to improve better than those of the basic components. A key objective in blend formulations is an improved property of base materials for enhanced performance to a wide range and durable applications of the matrix. The swelling power has been related to the associative binding within the polymeric unit and apparently, the strength and character of the micellar network is related to the amylaze content. Low amylase content is reported to produce high swelling power [21]. This behavior is instructive and may guaranty product stability during extreme whether conditions.

Sensory Evaluation

The mean score for odor and appearance of the base materials and formulations are shown in table 6. The base materials had average odor values of 0.16 and 0.1 respectively, almost an odorless rating. The odor levels of the formulations ranged from 1.00 – 1.66 and showed no significant difference (P>0.05). Generally odor ratings of all the formulations were judged as very faint offensive.

The mean values for appearance were significantly different (P>0.05). F_9 (5.66) as the highest, while F_5 (4.16) was the lowest. Although panelist varied in their assessment, which ranged between neutral to moderately liked, none of the formulations was disliked by the panelists. Odor assessment received similar trend. The overall acceptability score findings showed that none of the formulations was objectionable, and therefore would impart little or no effect when used in odor treatment to stabilize solid or liquid waste in the environment, for example.

Table 6: sensory evolution of base materials

Materials	**Color and appearance**	**Odor**
A	5.25	0.1
B	6.00	0.16

Table7: Sensory evolution of Matrix

Matrix	Color and appearance	Odor
F1	5.50	1.66
F2	5.16	1.00
F3	4.83	1.00
F4	4.83	1.16
F5	4.16	1.33
F6	4.80	1.00
F7	5.16	1.00
F8	4.33	1.00
F9	5.66	1.33

Sensitivity to microbial life

Effects of the matrix on microbial life were also tested. The results showed excellent sensitivity to a broad spectrum of microorganisms. They were highly sensitive to: e-coli, streptococcus, staphylococcus, Roteus vulgaris, and candida albican.

CONCLUSION

The formulation and preliminary investigations of the matrices exhibited excellent physical and chemical properties and show promise of a wide range of applications. Although all of the formulations improved greatly, F2 and F7 were at overall optimum levels. They will be potential materials for inhibiting odor causing microbial growth and therefore useful for waste water stabilization and control of animal waste odors.

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