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Effect of different extraction methods on starch production from millet flour and their reflection on morphological properties with nanotechnology-driven structural modification and nanoscale characterization

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This study investigated the effects of aqueous, acidic, and alkaline extraction methods on starch isolated from foxtail millet flour prepared at three hydration levels (12.2%, 15%, and 16%). Millet flour is produced using a Bühler laboratory mill with three breaking and two reduction stages, followed by starch extraction using the three solution systems. Alkaline extraction with 0.3% sodium hydroxide produced the highest starch yields, particularly for Bk 0.3% (15% hydration) and Ck 0.3% (16% hydration), achieving 63.63% and 63.23%, respectively, with only minor reductions in amylose content (17.5% and 19.2%). In contrast, acid extraction using 0.3% sodium acetate at 12.2% and 15% hydration resulted in lower yields (42.40% and 42.60%) while maintaining comparable amylose levels (≈18%). The lowest starch recoveries are observed for aqueous extraction, with yields of 30.39%, 33.50%, and 31.05% for Aw, Bw, and Cw samples, respectively. Increasing grain hydration from 12.2% to 16% enhanced starch yield and induced pronounced morphological changes, particularly under alkaline conditions, demonstrating the strong influence of hydration and extraction chemistry on starch recovery efficiency. Furthermore, nanotechnology-based approaches enable precise control of starch granule structure at the nanoscale, leading to enhanced surface area, improved functional properties, and the development of starch-derived nanomaterials suitable for advanced food, pharmaceutical, and industrial applications.

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Keywords: Morphological; Nanotechnology; Nanoscale structure; Starch; Nanomodification.

1. INTRODUCTION

Starch is the predominant component of millet grains, accounting for approximately 62.8–70.5% of the total grain mass, depending on genotype [1]. As the second most abundant polysaccharide in nature after cellulose, starch is an economical and essential energy source due to its amylose and amylopectin fractions [2]. Chemically, starch is composed of α-D-glucose units linked by α-1,4 and α-1,6 glycosidic bonds, forming linear amylose and highly branched amylopectin polymers. Amylose typically consists of 500–600 glucose units with minimal branching, whereas amylopectin has a much higher degree of polymerization (10,000–100,000 units) and extensive α-1,6 branching [3, 4]. The functional properties of starch—such as gelatinization, thickening, adhesion, and water solubility—are strongly influenced by the amylose-to-amylopectin ratio and their molecular organization within starch granules. Millet starch contains 17.03–23.30% amylose, classifying it as intermediate- to high-amylose starch [5]. In addition to carbohydrates, millet starch contains minor components, including ash (≈1.06%), protein (≈0.58%), and lipids (<1%), which significantly influence starch functionality and purity [6, 7]. Lipids can form amylose–lipid inclusion complexes that limit starch swelling and gelatinization but enhance stability in baked products, extending shelf life and reducing staling [8]. Starch extraction efficiency is influenced by millet hydration and the choice of extraction medium. Soaking softens grain structure, enhances water absorption, and facilitates starch release by disrupting starch–protein matrices [9]. Wet milling using water, acidic, or alkaline solutions is commonly employed, with alkaline leaching proving most effective for protein removal and amylose enrichment [10, 11]. Acid treatments often retain higher protein residues, whereas alkaline methods yield purer starch with modified granule surfaces, as confirmed by SEM studies [12]. High-amylose millet starches are therefore particularly attractive for developing functional foods, baked goods, and low-glycemic formulations [13]. Recent developments in nanotechnology have significantly enhanced the understanding and manipulation of starch structure at the nanoscale. Nanoscale modification techniques allow the transformation of native starch granules into functional nanostructured materials with improved physicochemical properties. These advances provide new opportunities for tailoring starch functionality for specialized applications in food systems, drug delivery, and biodegradable materials.

2. METHODS AND METHODS

Millet grains used: The millet grains for the 2023 marketing season are obtained from local markets. The grain variety is identified, and the millet grains are then cleaned of impurities using a grain cleaner.

2.1. Grain purification

Grain purification and separation from impurities based on grain size using the NSP grain purifier supplied by CHOPIN.

2.2. Grain tempering process

The samples are moistened with ordinary tap water at room temperature by adding a quantity of water to the containers holding the millet samples. The amount of water added (ml) is calculated according to the following equation [14-17]:

$$Amount\ of\ water\ added\ (ml) = \left(\frac{Model\ humidity - 100}{(Required\ humidity - 100) - 1}\right) \times Model\ weight \quad (1)$$

After hydration, the grains are left to stand for 24 hours, stirred several times. Hydration is applied in a single batch to the coded samples: (A) no hydration (direct milling), (B) 15% hydration, and (C) 16% hydration.

2.3. Grain milling

The samples are milled using a Buhler Laboratory Mill, as specified in AACC 26-31.01 (2010). The extraction rate (flour yield) is calculated using the following equation [18-22]

$$Productivity\ \% = \frac{Flour\ weight}{Clean\ dry\ grain\ weight} \times 100 \quad (2)$$

2.4. Methods of starch extraction

Starch is extracted from millet flour using the three methods mentioned by [23, 24] with some modifications.

1. Water extraction of Starch from Millet Flour

100 g of millet flour from the following treatments (no hydration (A), 15% hydration (B), 16% hydration (C)) is soaked in 250 mL of distilled water for 24 hours at 4°C. The soaked flour is then rapidly mixed for 2 minutes. The mixture is washed through a No. 100 (150 mm) and No. 200 (75 mm) American sieve. The product is filtered and centrifuged at 6000 × g for 15 minutes, and the resulting supernatant is filtered. The starch granules are washed with distilled water. The method is modified by passing the mixture through a No. 200 sieve, then pouring the mixture, after removing the supernatant (centrifugation), into dishes and drying at room temperature for 24 hours.

2. The Second Method (Alkaline Soaking Method)

The second method is determined based on the method described by Wang and Wang (2001), with minor modifications. One hundred g of millet flour is soaked in 250 mL of sodium hydroxide (NaOH) at concentrations of 1 g/kg, 2 g/kg, and 3 g/kg for 18 hours at 4°C. The soaked flour is then rapidly mixed for 2 minutes, filtered through 150 mm and 75 mm sieves respectively, and centrifuged at 6000 × g for 10 minutes. The lower starch layer is remixed and washed with 1 g/kg of sodium hydroxide and water, then neutralized with 1 M hydrochloric acid to a pH of 6.5 and centrifuged. The starch is then washed with distilled water and dried at room temperature for 24 hours.

3. The third method (acid soaking method)

Starch is isolated from millet flour according to the procedure of Adkins and Greenwood (1966), with slight modifications. One hundred g of millet flour is soaked in 250 mL of aqueous sodium acetate at concentrations of 1 g/kg, 2 g/kg, and 3 g/kg for 12 hours at 4°C. The soaked flour is then rapidly mixed for two minutes, filtered through 150 mm and 75 mm sieves respectively, and centrifuged at 6000 x g for 15 minutes. The supernatant is discarded, and the starch granules are washed with 1 M sodium chloride solution and centrifuged at 6000 x g for 10 minutes. Finally, the starch is dried at room temperature for 24 hours.

(a) The chemical properties of common millet flour are determined according to AACC (2000)

Moisture content is measured by drying 3 grams of flour in an oven at 130°C until a constant weight is reached (Method 15-44A). Ash content is determined by burning at 550°C until a constant weight is reached (Method 08-01). Nitrogen content is determined using the Kjeldal method. Using a coefficient of 6.25, it is converted to protein (Method A46-11). Lipid content is determined using the Soxhlet method (Method 10-30).

(b) Fiber Content Estimation

The fiber content of millet starch samples is determined using a gravimetric dry-mass method. The powdered millet starch samples are homogenized and oven-dried at 105 °C to constant weight. A known quantity of each dried sample is weighed and gently disaggregated to ensure uniformity. The samples are passed through standard sieves to remove non-fibrous particles. The retained fraction is washed with deionized water to eliminate fine residues, leaving the fibrous material. This fraction is oven-dried again at 105 °C to constant weight and weighed. The fiber content is calculated using the formula [24-28]:

$$\text{Fiber (\%)} = \frac{\text{dry mass of fibrous fraction}}{\text{initial dry mass of sample}} \times 100 \quad (3)$$

(c) Calculating Starch Yield and Recovery

For all methods, starch yield is determined according to [29-31],

where

$$\%\,Yield = \frac{(Dry\ weight\ of\ starch\ recovered\ from\ extraction \times 100)}{Dry\ weight\ of\ flour\ or\ whole\ corn\ kernels\ (g)} \quad (4)$$

Starch recovery from extraction is calculated using the dry weight of corn kernels as follows [32-35]:

$$\%\,Yield = \frac{(Percentage\ of\ starch\ yield \times 100)}{Total\ starch\ in\ corn\ kernels} \quad (5)$$

(d) Enzymatic Determination of Amylose Content

The starch samples are completely dispersed by heating in dimethyl sulfoxide (DMSO). Lipids are removed by precipitating the starch in ethanol and recovering the precipitated starch. After dissolving the precipitated sample in acetate/salt solution, amylopectin is specifically precipitated by the addition of Con A and removed by centrifugation. Amylose, in a portion of the upper liquid, is enzymatically degraded to D-glucose, which is then analyzed using glucose oxidase/peroxidase reagent. Total starch, in a separate portion of the acetate/salt solution, is similarly degraded to D-glucose and quantified chromatically by glucose oxidase/peroxidase (GOPOD). The amylose concentration in the starch sample is estimated as the GOPOD uptake ratio at 510 nm of the upper liquid of the precipitated Con A sample to the total starch sample.

This procedure applies to all pure starch samples and to cereal flour [36-40].

$$Amylose\ \% = \frac{Upper\ fluid\ absorbance\ (CON\ A)}{Total\ starch\ absorbance \times 66.8} \quad (6)$$

2.5.Morphological characteristics

Morphological characteristics are analyzed using SEM. Ethanol is used to prepare a 1% starch solution for recording the microscopic images. The starch solution is transferred to a metal tube coated with Au-Pd (60:40). The samples are evaluated at a 10 kV accelerating potential, and a magnification of 2500× is used during microscopy.

2.6. Statistical analysis

The data are analyzed using the analysis of variance (ANOVA) test, and the differences between the means are compared in terms of statistical significance ($p < 0.05$) using SPSS version 27.

2.7. Nanotechnology-based structural analysis

Nanotechnology-based analytical approaches are considered to better understand the structural transformation of starch granules during extraction. Techniques such as high-resolution scanning electron microscopy (SEM), nanoscale particle size analysis, and surface morphology evaluation

enable detailed characterization of starch nanostructures. These approaches provide insight into the formation of nanoporous networks and nanofibrous structures resulting from chemical treatments.

4. RESULT AND DISCUSSION

Table 1 presents the effect of different extraction methods (aqueous, acidic, and alkaline) on the chemical composition and starch yield of millet flour obtained by direct milling at 12.2% grain hydration. Significant differences ($p \leq 0.05$) among treatments are indicated by different superscript letters.

Aqueous extraction (Aw) produced the lowest starch yield (30.39%), indicating limited disruption of the starch–protein matrix despite low protein content (0.9%) and moderate amylose levels (18.7%). Acid extraction increased starch yield with concentration, peaking at 0.2% sodium acetate (48.35%), before declining at 0.3% due to partial starch solubilization. Acid treatments showed reduced protein removal and slight amylose loss at higher concentrations. Alkaline extraction is the most effective, with starch yield increasing to 61.40% at 0.3% NaOH, attributed to efficient protein dissolution. Although minor increases in protein and fat are observed at higher alkali levels, alkaline treatments yielded higher total starch and carbohydrates overall, confirming their superiority for starch recovery from millet flour.

Table 1 Effect of different extraction methods on starch yield% from millet flour (grains hydrated at 12.2 %)(direct milling).

Treatment name	Moisture content %	Ash Content (%)	Crude Fiber Content (%)	Protein Content (%)	Fat Content (%)	Total Carbohydrates %	Total starch %	Amylose % enzymatic	yield%
Aw	12.8 ± 0.3d	0.9 ± 0.1c	2.3 ± 0.2ab	0.9 ± 0.1a	1.4 ± 0.1b	81.7 ± 1.2ab	67.5 ± 1.0a	18.7 ± 0.5c	30.39 ± 1.2a
Ac0.1%	12.5 ± 0.3cd	0.8 ± 0.1b	2.5 ± 0.2bc	1.5 ± 0.1b	1.5 ± 0.1c	81.2 ± 1.1a	68.0 ± 1.1ab	17.4 ± 0.4b	42.56 ± 1.5c
Ac0.2%	11.8 ± 0.3b	0.7 ± 0.1a	2.3 ± 0.2ab	1.8 ± 0.1c	1.2 ± 0.1a	82.2 ± 1.3bc	69.5 ± 1.2c	16.9 ± 0.4ab	48.35 ± 1.8d
Ac0.3%	13.0 ± 0.4d	0.9 ± 0.1c	2.6 ± 0.2c	2.4 ± 0.2e	1.4 ± 0.1b	79.7 ± 1.1a	67.8 ± 1.0a	18.0 ± 0.5bc	42.40 ± 1.5c
Ak0.1%	12.0 ± 0.3bc	0.6 ± 0.1a	2.4 ± 0.2abc	1.2 ± 0.1a	1.3 ± 0.1ab	82.5 ± 1.3c	68.5 ± 1.1b	17.1 ± 0.4b	38.62 ± 1.4b
Ak0.2%	11.5 ± 0.3a	0.8 ± 0.1b	2.2 ± 0.2a	1.5 ± 0.1b	1.1 ± 0.1a	82.9 ± 1.4c	70.0 ± 1.3d	19.0 ± 0.6d	55.98 ± 2.1e
Ak0.3%	12.3 ± 0.3c	0.7 ± 0.1a	2.5 ± 0.2bc	2.1 ± 0.2d	1.6 ± 0.1d	80.8 ± 1.2ab	68.0 ± 1.1ab	17.2 ± 0.4b	61.40 ± 2.3f

A=12.2% unhydrated (direct mill), w = water extraction, k=base extraction, c=acid extraction.

Table 2 summarizes the influence of aqueous, acidic, and alkaline extraction methods on the chemical composition and starch yield of millet flour hydrated to 15%. Statistically significant differences ($p \leq 0.05$) are observed among treatments.

Aqueous extraction (Bw) produced a low starch yield (33.50%), reflecting limited disruption of starch–protein interactions despite low protein content (0.6%) and moderate amylose levels (16.5%). Acid extraction showed concentration-dependent behavior, with the highest yield at 0.1% sodium acetate

(43.42%). Increasing acid concentration reduced yield at 0.2% due to partial starch solubilization, while a slight recovery at 0.3% is accompanied by increased protein content (2.7%), indicating reduced purity. Alkaline extraction is the most effective at this hydration level, with starch yield rising to 63.63% at 0.3% NaOH. This improvement is attributed to efficient protein dissolution and starch release, although higher alkali levels caused minor increases in residual protein [41-45].

Table 2 Effect of different extraction methods on starch yield% from millet flour (grains hydrated at 15 %).

Treatment name	Moisture content %	Ash Content (%)	Crude Fiber Content (%)	Protein Content (%)	Fat Content (%)	Total Carbohydrates %	Total starch %	Amylose % enzymatic	yield%
Bw	12.2 ± 0.3c	0.7 ± 0.1b	2.8 ± 0.2d	0.6 ± 0.1a	1.5 ± 0.1c	82.2 ± 1.3bc	68.0 ± 1.1ab	16.5 ± 0.4a	33.50 ± 1.3a
Bc0.1%	11.9 ± 0.3b	0.6 ± 0.1a	2.4 ± 0.2ab	1.5 ± 0.1b	1.3 ± 0.1b	82.3 ± 1.3c	69.0 ± 1.2bc	17.5 ± 0.4b	43.42 ± 1.6c
Bc0.2%	12.1 ± 0.3bc	0.8 ± 0.1c	2.7 ± 0.2cd	2.1 ± 0.2d	1.5 ± 0.1c	80.8 ± 1.2ab	66.5 ± 1.0a	17.0 ± 0.4ab	39.40 ± 1.5b
Bc0.3%	12.4 ± 0.3c	0.7 ± 0.1b	2.6 ± 0.2bc	2.7 ± 0.2e	1.2 ± 0.1a	80.4 ± 1.2a	68.2 ± 1.1ab	18.1 ± 0.5c	42.60 ± 1.6bc
Bk0.1%	11.7 ± 0.3a	0.5 ± 0.1a	2.5 ± 0.2abc	0.9 ± 0.1a	1.4 ± 0.1bc	83.0 ± 1.4d	70.1 ± 1.3d	17.6 ± 0.4b	38.50 ± 1.4b
Bk0.2%	12.6 ± 0.3d	0.9 ± 0.1d	2.4 ± 0.2ab	1.5 ± 0.1b	1.3 ± 0.1b	81.3 ± 1.2ab	67.9 ± 1.0a	19.0 ± 0.6d	50.70 ± 1.9d
Bk0.3%	11.4 ± 0.3a	0.6 ± 0.1a	2.3 ± 0.2a	2.1 ± 0.2d	1.1 ± 0.1a	82.5 ± 1.3c	70.5 ± 1.3d	17.5 ± 0.4b	63.63 ± 2.4f

B=hydrated 15%, w = water extraction, k=base extraction, c=acid extraction.

Table 3 illustrates the effect of extraction method on starch yield and composition from millet flour hydrated at 16%. Aqueous extraction (Cw) resulted in the lowest starch yield (31.05%), despite low protein (0.6%) and relatively high total starch (70.0%), indicating that water alone is insufficient to disrupt starch–protein associations even at higher hydration.

Acid extraction (Cc) significantly improved starch recovery compared to water extraction. The highest acid-derived yield is observed at 0.2% sodium acetate (55.37%), accompanied by increased total starch (70.8%) and amylose content (19.0%). However, further increasing acid concentration to 0.3% reduced yield (46.41%) while markedly increasing protein content (2.4%) and amylose (21.5%), suggesting partial starch degradation and reduced purification efficiency at higher acid strength [46-50]. Alkaline extraction (Ck) is the most effective method at this hydration level. Starch yield increased consistently

with NaOH concentration, reaching a maximum of 63.23% at 0.3%, the highest among all treatments. This enhancement is attributed to efficient protein solubilization and disruption of the starch–protein matrix. Although higher alkali concentrations resulted in slightly elevated protein and fat contents, total carbohydrate levels remained high, indicating good starch purity [51-55].

Table 3 Effect of different extraction methods on starch yield% from millet flour (grains hydrated at 16 %).

Treatment name	Moisture content %	Ash Content (%)	Crude Fiber Content (%)	Protein Content (%)	Fat Content (%)	Total Carbohydrates %	Total starch %	Amylose % enzymatic	yield%
Cw	12.1 ± 0.3c	0.7 ± 0.1b	2.4 ± 0.2a	0.6 ± 0.1a	1.3 ± 0.1b	82.9 ± 1.4d	70.0 ± 1.3d	18.9 ± 0.5d	31.05 ± 1.2a
Cc0.1%	12.0 ± 0.3bc	0.8 ± 0.1c	2.5 ± 0.2ab	1.5 ± 0.1b	1.4 ± 0.1c	81.8 ± 1.3c	69.5 ± 1.2cd	18.0 ± 0.4c	51.15 ± 1.9d
Cc0.2%	11.6 ± 0.3a	0.5 ± 0.1a	2.2 ± 0.2a	1.8 ± 0.1c	1.2 ± 0.1a	82.7 ± 1.4d	70.8 ± 1.4d	19.0 ± 0.6d	55.37 ± 2.1e
Cc0.3%	12.3 ± 0.3d	0.7 ± 0.1b	2.6 ± 0.2b	2.4 ± 0.2d	1.3 ± 0.1b	80.7 ± 1.2b	68.3 ± 1.1b	21.5 ± 0.7e	46.41 ± 1.7c
Ck0.1%	11.8 ± 0.3ab	0.6 ± 0.1a	2.4 ± 0.2a	1.2 ± 0.1a	1.1 ± 0.1a	82.9 ± 1.4d	69.2 ± 1.2c	18.6 ± 0.5cd	50.37 ± 1.9d
Ck0.2%	12.5 ± 0.3d	0.9 ± 0.1d	2.7 ± 0.2b	1.5 ± 0.1b	1.5 ± 0.1c	80.9 ± 1.2b	66.7 ± 1.0a	20.3 ± 0.6e	52.67 ± 2.0de
Ck0.3%	11.9 ± 0.3ab	0.8 ± 0.1c	2.5 ± 0.2ab	2.1 ± 0.2c	1.4 ± 0.1bc	81.3 ± 1.2bc	68.8 ± 1.1bc	19.2 ± 0.6d	63.23 ± 2.4f

C= hydrated 16%, w = water extraction, k=base extraction, c=acid extraction.

Productivity data showed significant differences ($p < 0.05$) among extraction treatments, as indicated by distinct superscript letters. Aqueous controls (Aw, Bw, and Cw) consistently formed the lowest statistical group, yielding only 30.39–33.50%, which reflects limited disruption of the starch–protein matrix. In contrast, alkaline extraction at 0.3% NaOH produced the highest starch yields across all hydration levels (61.40–63.63%), representing the highest statistical group and confirming sodium hydroxide as the most effective extraction medium. Acid extraction resulted in intermediate, concentration-dependent yields (38.62–55.98%), with yields declining above 0.2% sodium acetate, suggesting saturation or inhibitory effects at higher acid concentrations. Total starch content showed limited variation (66.7–70.8%) and generally followed yield trends, while amylose content ranged from 16.5% to 21.5%, with acetate treatments tending to reduce amylose compared to aqueous extraction. Protein content increased with increasing acetate concentration, indicating reduced purification efficiency. Increasing grain hydration enhanced starch recovery primarily under alkaline conditions, whereas hydration had a weaker effect on acid extraction. Moisture and ash contents remained relatively stable, demonstrating that extraction chemistry exerted a stronger influence than hydration level. Overall, high-concentration alkaline treatments produced starch with superior yield, high purity, moderate amylose content, and low fiber levels, making them optimal for efficient millet starch extraction [56-60].

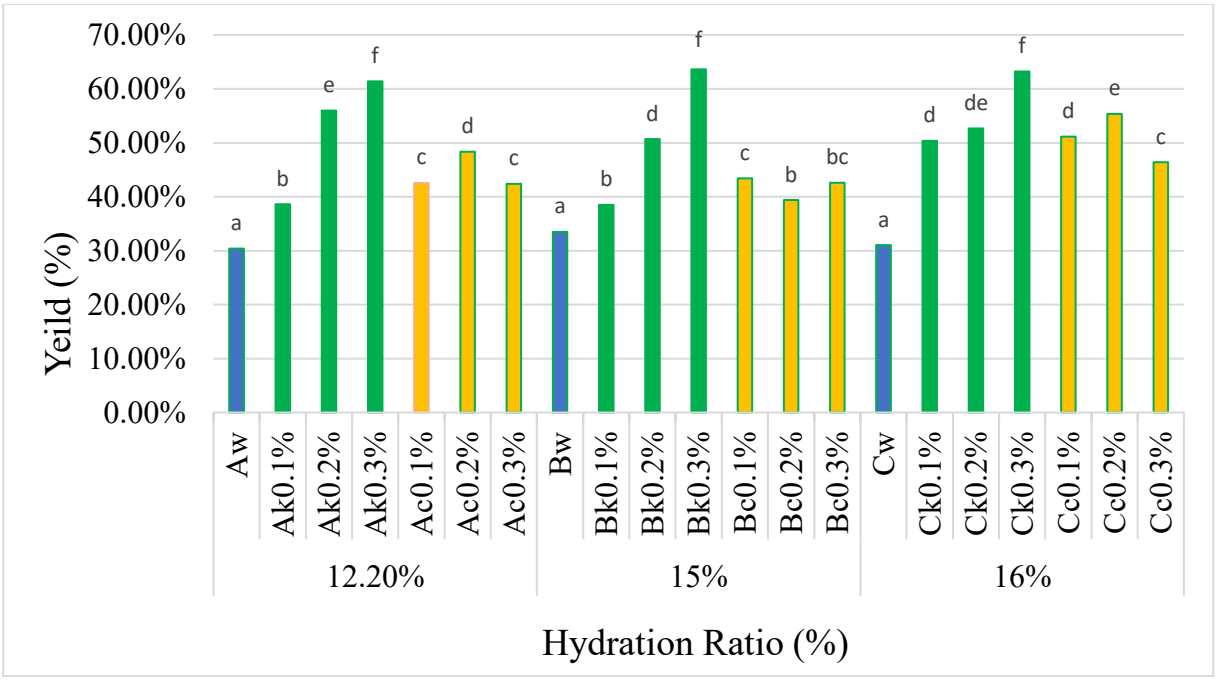

Figure 1 The effect of different extraction methods on the percentage of starch production from millet flour (for grains moistened at different rates before the milling process).

4.1.Morphological characteristics

Native millet starch extracted by aqueous treatment (AW) at 12.2% hydration exhibits well-defined polyhedral granules (1–3 μm) with smooth, intact surfaces at 75,000× magnification. Sharp edges, uniform electron density, and clearly preserved growth rings (15–20 nm) confirm an intact semicrystalline organization with alternating amorphous and crystalline lamellae, characteristic of native starch. Acid extraction induces concentration-dependent morphological changes. At 0.1% sodium acetate (Ac 0.1), intact granules coexist with eroded and agglomerated particles showing surface craters and pores (100–500 nm), indicating partial hydrolysis of amorphous regions. At 0.2% (Ac 0.2), approximately 60–70% of granules display surface pitting and channel formation (100–300 nm), with cracked edges revealing lamellar structures and partial crystalline preservation. At 0.3% (Ac 0.3), granules are almost completely dissociated into a porous nanonetwork of 100–300 nm fragments. In contrast, alkaline extraction produces more selective and ordered modifications: mild nanoscale etching at 0.1% NaOH, oriented nanochannels at 0.2%, and a highly porous, foam-like nanostructure at 0.3%, composed of amylose fibrils and residual crystalline clusters (Figure 2) [61-66].

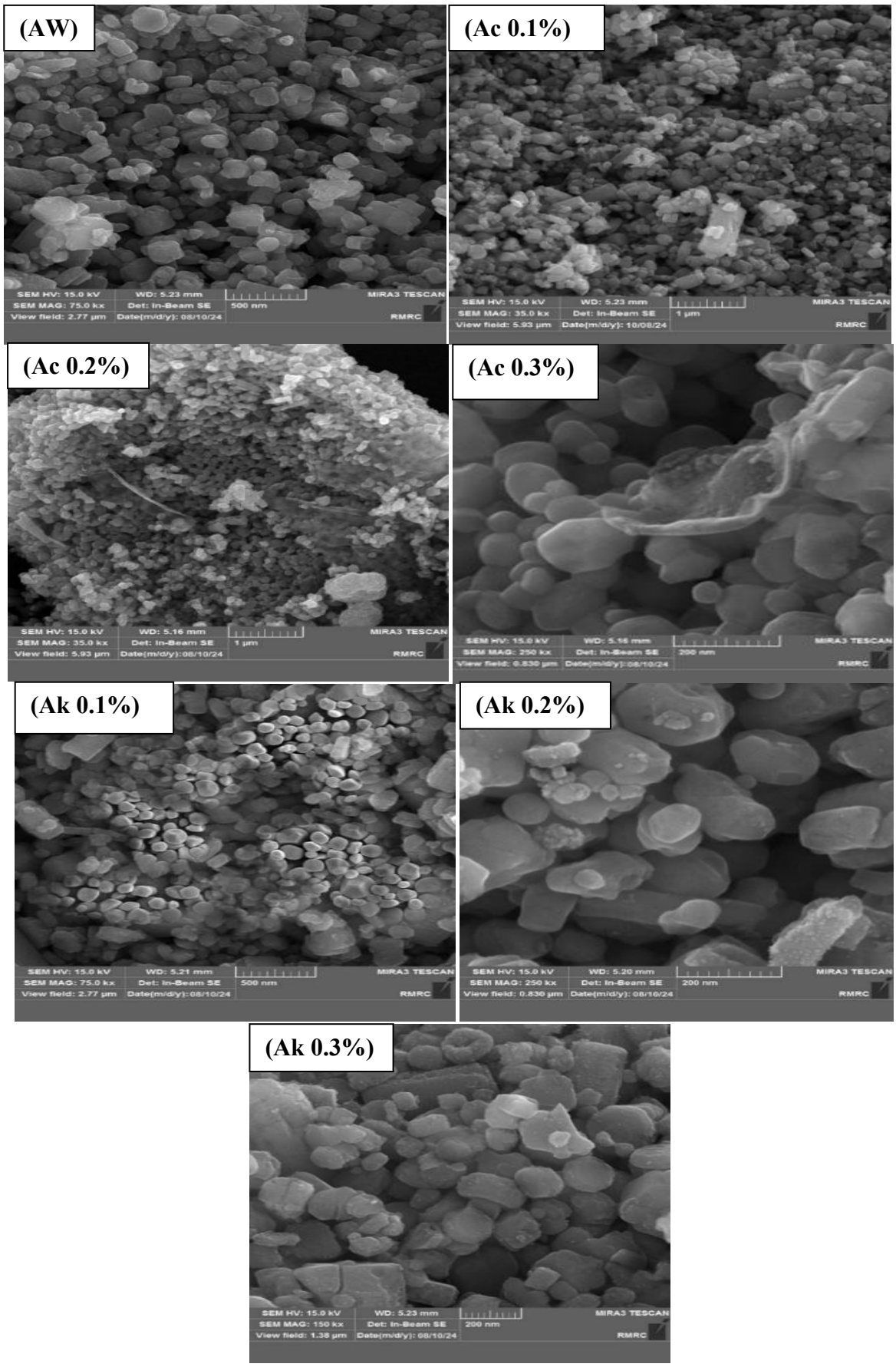
(AW)
(Ac 0.1%)
(Ac 0.2%)
(Ac 0.3%)
(Ak 0.1%)
(Ak 0.2%)
(Ak 0.3%)

Figure 2 Scanning electron micrographs illustrating the progressive morphological changes in millet starch extracted at 12.2% hydration using aqueous, acidic, and alkaline methods. Native starch (AW) exhibits intact polyhedral granules with smooth surfaces and well-preserved semicrystalline organization.

Starch extracted from millet flour at 15% hydration exhibited clear, concentration-dependent morphological changes depending on extraction chemistry. The aqueous-extracted sample (BW) showed mostly intact polygonal granules (5–12 μm) with smooth surfaces and minimal surface pitting (<5%), indicating gentle extraction and preservation of native starch structure. Acid extraction at 0.1% sodium acetate (Bc 0.1) induced moderate surface erosion, with 30–40% of granules showing pits and channels (200–500 nm) while largely retaining granule integrity. At 0.2% (Bc 0.2), extensive and uniform surface porosity developed, forming interconnected channels (50–200 nm) while preserving overall granule shape, suggesting selective hydrolysis of amorphous regions. At 0.3% (Bc 0.3), granules are completely transformed into a nanoporous matrix of interconnected starch fragments (20–100 nm), eliminating native gelling behavior. Alkaline extraction produced more pronounced and ordered modifications: Bk 0.1 caused uniform surface etching with partial fragmentation, Bk 0.2 generated mixed populations of porous granules and nanofragments, and Bk 0.3 resulted in complete granule dissociation into a three-dimensional nanostructured network, indicating a transition from modified starch to starch-based nanomaterials (Figure 3) [67-70].

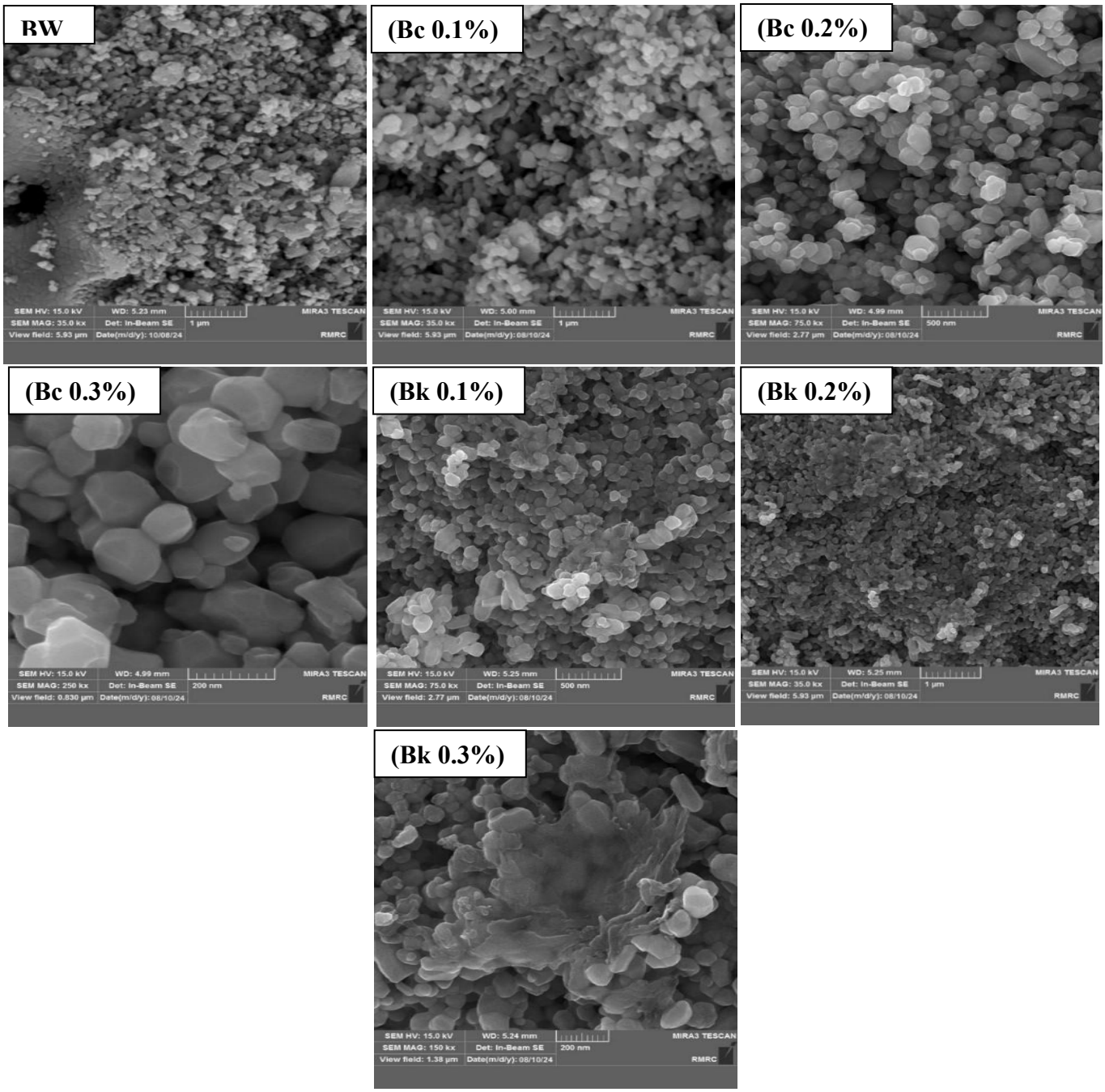

Figure 3 Scanning electron micrographs illustrating the morphological evolution of millet starch extracted at 15% hydration using aqueous, acidic, and alkaline treatments.

Figure 4 presents the morphological evolution of millet starch extracted at a 16% hydration level under aqueous, acidic, and alkaline conditions. Water-extracted starch (Cw) exhibits well-preserved native granules with smooth, angular morphology (4–10 μm), clear growth rings, and minimal surface defects, confirming intact semicrystalline organization and preservation of native thermal behavior. Acid extraction induces concentration-dependent surface modification: mild, uniform nano-pitting at 0.1% (Cc 0.1) selectively targets amorphous regions while retaining granule integrity; at 0.2% (Cc

0.2), heterogeneous structures emerge with porous granules, partial fragmentation, and exposed crystalline platelets; and at 0.3% (Cc 0.3), granules are fully dissociated into a fibrous nanonetwork with embedded crystalline domains, enhancing adsorption but reducing gelling capacity. In contrast, alkaline extraction produces more pronounced and ordered restructuring. Progressive NaOH treatment transforms granules from deeply pitted, terraced structures (Ck 0.1) to bimodal porous–nanocrystalline systems (Ck 0.2), culminating at 0.3% (Ck 0.3) in a hierarchical nanofiber–crystalline sheet network with interconnected pores. Overall, Figure 4 highlights the superior ability of alkaline extraction to generate highly ordered, high-surface-area starch-derived nanomaterials [71-75].

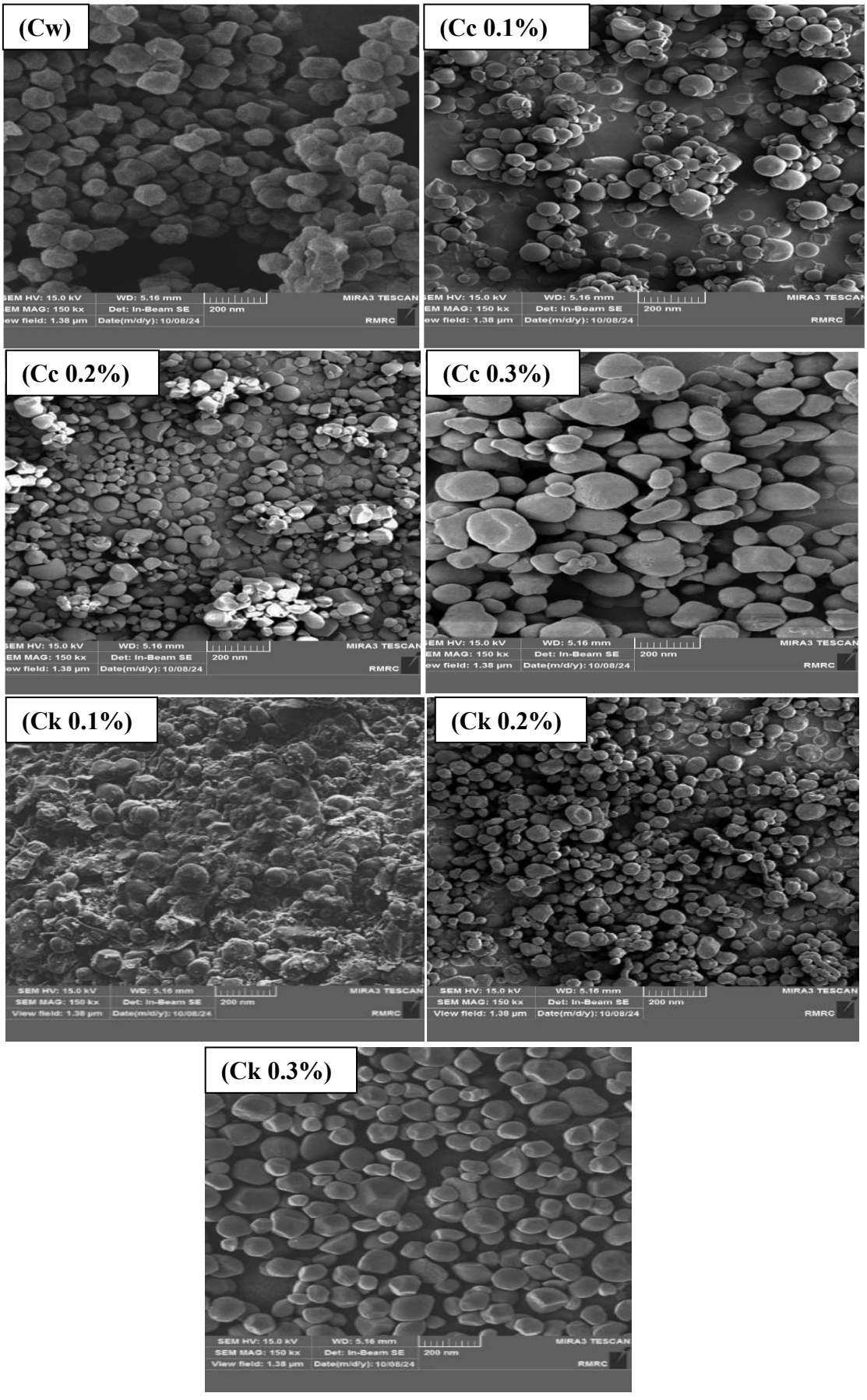
(Cw)
(Cc 0.1%)
(Cc 0.2%)
(Cc 0.3%)
(Ck 0.1%)
(Ck 0.2%)
(Ck 0.3%)
SEM HV: 15.0 kV
WD: 5.16 mm
MIRA3 TESCAN
SEM MAG: 150 kx
Det: In-Beam SE
200 nm
View field: 1.38 µm
Date(m/d/y): 10/08/24
RMRC

Figure 4 Scanning electron micrographs illustrating the morphological transformations of millet starch extracted at 16% hydration using aqueous, acidic, and alkaline treatments.

4.2.Effect of nanotechnology on starch nanostructure and functionality

The application of nanotechnology principles in starch extraction reveals significant structural transformation at the nanoscale. Alkaline and acid treatments lead to the formation of nanoporous networks and nanofibrous structures, increasing surface area and enhancing functional performance [76-80]. These nanoscale modifications improve water absorption, solubility, and adsorption properties, demonstrating the potential of starch as a nanostructured material for advanced applications [81, 82]. Table 3 presents a comparison between conventional starch and nanostructured starch, demonstrating significant improvements in surface area, adsorption capacity, and functional performance due to nanotechnology-driven modification [83].

Table 3 Comparison between conventional starch and nanostructured starch produced by different extraction methods, highlighting nanoscale structural transformation and enhanced functional properties.

Parameter	Conventional Starch	Nano-Structured Starch
Structure	Granular	Nanofibrous / Nanoporous
Surface Area	Low	High
Particle Size	Micron scale	Nano scale
Adsorption Capacity	Moderate	High
Functional Performance	Standard	Enhanced

Figure 5 presents the transformation of native starch granules into nanostructured materials, highlighting the formation of nanoporous networks and nanofibrous structures under chemical treatments.

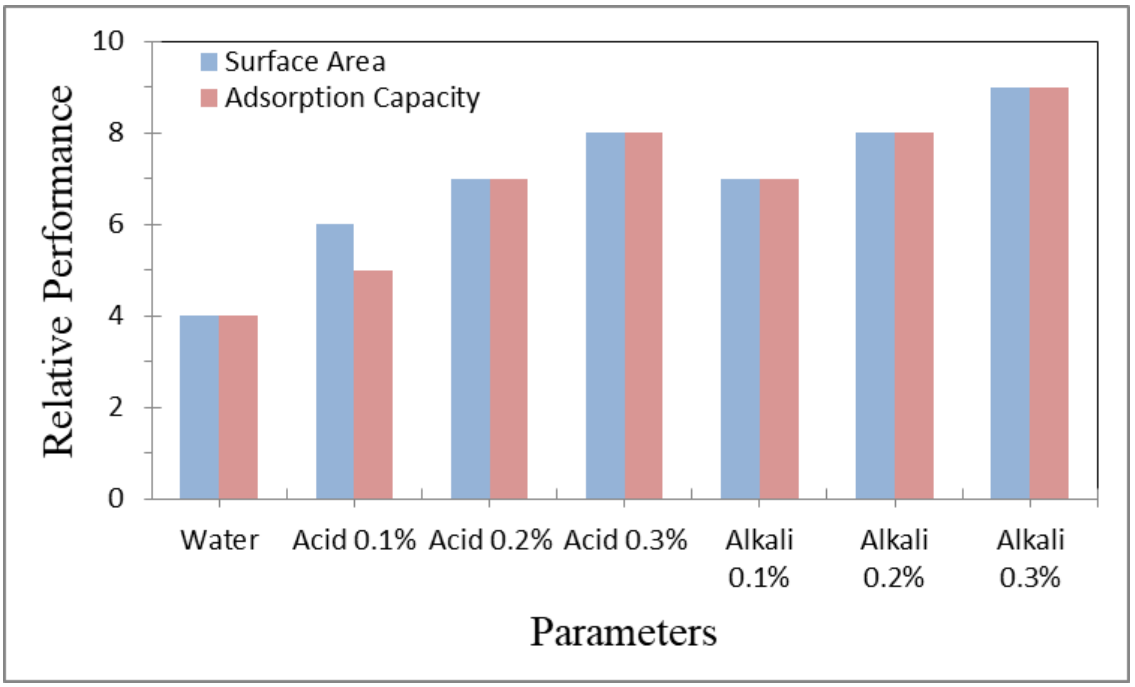

Figure 5 Nanotechnology-induced transformation of starch granules into nanoporous and nanofibrous structures under different extraction conditions.

5. CONCLUSIONS

The three extraction methods generate distinctly different starch morphologies. Aqueous extraction (AW) preserves the native granular structure, while sodium acetate (Ac 0.3) produces nano-fragmented starch with a porous architecture. In contrast, sodium hydroxide (Ak 0.3) causes complete loss of the original polyhedral granules, yielding foam-like biomechanical structures. These differences strongly influence functionality: AW starch behaves like native starch, whereas the porous nanostructure of Ac 0.3 starch is promising for adsorption applications. For starch extracted from millet flour, alkaline treatments induce more pronounced structural modifications than acid treatments at equivalent concentrations. While 0.1% acid causes mild surface corrosion, 0.1% base generates substantial porosity. At 0.3%, alkaline treatment fully dissociates granules, whereas acid treatment leaves residual structures. Alkaline extraction selectively disrupts amorphous regions, producing cleaner fracture surfaces and larger surface areas. At higher hydration (16%), base treatments promote multilevel structural reorganization and yield starch-derived nanomaterials with properties distinct from conventional modified starches, highlighting their potential in advanced food, pharmaceutical, and materials applications. The integration of nanotechnology into starch extraction processes provides a powerful framework for transforming conventional starch into advanced nanostructured materials. Nanoscale modifications enhance surface area, adsorption capacity, and functional properties, expanding the potential applications of millet starch in food, pharmaceutical, and industrial fields. Future studies should focus on optimizing nano-extraction techniques to further improve starch performance and functionality.

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