

Ciência e Tecnologia

Para o Desenvolvimento
Ambiental, Cultural
e Socioeconômico

Xosé Somoza Medina
(organizador)

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 EDITORA
ARTEMIS
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PRÓLOGO

Un nuevo volumen de la serie ***Ciência e Tecnologia para o Desenvolvimento Ambiental, Cultural e Socioeconômico***, el noveno ya, nos permite volver a reflexionar sobre los avances científico-tecnológicos y la transferencia de conocimientos a la sociedad. La ciencia y la tecnología ocupan hoy un lugar central en la comprensión y transformación de los distintos ámbitos de la vida contemporánea, pero tan importante es el avance en sí como la creciente posibilidad de difusión de esas innovaciones. El desarrollo actual no se limita a la creación de nuevos dispositivos, procesos o sistemas, sino que se manifiesta también en la capacidad de analizar problemas complejos, optimizar recursos, generar soluciones sostenibles, fortalecer la formación científica y ampliar las formas de interacción entre conocimiento, sociedad y desarrollo. Este libro es un buen ejemplo de esto último.

El presente volumen, ***Ciência e Tecnologia para o Desenvolvimento Ambiental, Cultural e Socioeconômico IX***, reúne investigaciones que reflejan esta diversidad de aplicaciones a la sociedad de diferentes avances científicos y técnicos. Los capítulos aquí presentados abordan problemas y aplicaciones diversas, pero comparten una misma orientación: la búsqueda de respuestas actuales a situaciones problemáticas fundamentadas desde la ingeniería, la innovación tecnológica, la ciencia de materiales, la energía, el ambiente, la inteligencia artificial, la formación científico-tecnológica, o la comunicación de la ciencia.

La obra se organiza en tres grandes ejes temáticos.

El primero, ***“Ingeniería, Metodologías y Procesos Tecnológicos”***, reúne seis trabajos vinculados con modelado dinámico, robótica, diagnóstico técnico mediante vibraciones, procesos de corte y manufactura, mejora de la productividad industrial, estudio de materiales y biopolímeros y metodología de investigación en ingeniería. Estos capítulos evidencian el papel de la tecnología en la modelización, experimentación y optimización de procesos y la importancia de los avances metodológicos en el desarrollo de soluciones aplicadas a contextos industriales y científicos.

El segundo eje, ***“Energía y Sostenibilidad”***, integra cinco investigaciones relacionadas con la generación de energía mediante microturbinas hidráulicas, el aprovechamiento de la energía de las mareas, la exposición a contaminantes derivados del uso de biomasa como fuente de energía, la importancia de las plantas medicinales en la dieta y en la cultura tradicional y los nuevos enfoques de la comunicación pública sobre energía nuclear. En conjunto, estos estudios muestran cómo la energía, cualquier tipo de energía, es un ámbito del conocimiento científico y técnico de importancia clave en

nuestra sociedad actual. La energía importa, más que nunca, y la ciencia y la tecnología pueden contribuir tanto al aprovechamiento responsable de los recursos naturales como a la comprensión de sus impactos ambientales y sociales.

El tercer eje, **“Formación y Comunicación en la era de la Inteligencia Artificial”**, amplía el campo de análisis hacia otro ámbito de total actualidad científica y tecnológica, la Inteligencia Artificial. Los seis trabajos reunidos en esta sección examinan el rendimiento académico a través de análisis estadísticos avanzados, el uso de podcasts como herramientas de desarrollo socioemocional docente, la aplicación de la inteligencia artificial en la gestión pública, las dificultades en el aprendizaje por la falta de pensamiento lógico-matemático en una muestra de estudiantes de ingeniería de sistemas computacionales, un modelo de análisis multivariante para evaluar el proceso de aprendizaje y el uso de mundos virtuales para mejorar las capacidades del profesorado. Este conjunto de contribuciones permite observar cómo las tecnologías digitales y la Inteligencia Artificial no solo modifican procedimientos técnicos, sino también las formas de enseñar, aprender y construir significado en el mundo académico.

La diversidad de enfoques presente en esta obra constituye uno de sus principales aportes. Lejos de entender la ciencia y la tecnología como campos aislados, los trabajos aquí reunidos muestran su carácter transversal y su capacidad para dialogar con cuestiones ambientales, educativas, sociales, productivas y culturales.

Esperamos que este volumen contribuya al intercambio de conocimientos, al fortalecimiento de nuevas investigaciones y a la difusión de experiencias y propuestas capaces de ampliar las posibilidades de desarrollo científico y tecnológico en diferentes contextos.

Xosé Somoza Medina

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SUMÁRIO

ENGENHARIA, METODOLOGIAS E PROCESSOS TECNOLÓGICOS

CAPÍTULO 1..... 1

ANÁLISIS DEL MODELO DINÁMICO DE UN MANIPULADOR ROBÓTICO INDUSTRIAL EN REFERENCIA AL DISEÑO DE SU SISTEMA DE CONTROL

Alejandro Armando Hossian

Roberto Carabajal

Carolina Hirschfeldt

Emanuel Maximiliano Alveal

 https://doi.org/10.37572/EdArt_2809263071

CAPÍTULO 2..... 16

APPLICATION OF VIBRATION EFFECTS IN TECHNICAL DIAGNOSTICS AND IDENTIFICATION

Vitalijs Beresnevics

 https://doi.org/10.37572/EdArt_2809263072

CAPÍTULO 3.....32

CORTE POR CIZALLAMIENTO DE UNA SILUETA CIRCULAR BAJO CONDICIONES DE MÍNIMO Y MÁXIMO MATERIAL

Pedro de Jesús García Zugasti

Enrique Alejandro Vázquez Hernández

José de Jesús Navarro Delgado

Yolanda Rodríguez Corpus

Juan Compeán Rodríguez

 https://doi.org/10.37572/EdArt_2809263073

CAPÍTULO 4..... 46

IMPLEMENTACIÓN DE METODOLOGÍA ERIM PARA INCREMENTO DE PRODUCCIÓN EN ÁREA DE ACABADO DE ROTORES

Enrique Alejandro Vázquez Hernández

Pedro de Jesús García Zugasti

Juan Gabriel Sandoval Granja

Gerardo García Liñán

Luis Antonio Renovato Rivera

 https://doi.org/10.37572/EdArt_2809263074

CAPÍTULO 5..... 58

ROLE OF THE INTERACTION AMYLOSE-NANOCELLULOSE ON THE RHEOLOGICAL PROPERTIES AND MOISTURE SORPTION OF MAIZE STARCH

Ignacia González

Makarena Jiménez

Paulo Díaz-Calderón

 https://doi.org/10.37572/EdArt_2809263075

CAPÍTULO 6..... 72

CONSTRUCCIÓN DEL MARCO TEÓRICO EN PROYECTOS DE INVESTIGACIÓN EN INGENIERÍA: REVISIÓN NARRATIVA Y ORIENTACIONES DIDÁCTICAS PARA ESTUDIANTES DE LICENCIATURA

Javier Montoya Ponce

Bertha Ivonne Sánchez Luján

María Teresa Martínez Acosta

María Guadalupe Amado Moreno

 https://doi.org/10.37572/EdArt_2809263076

ENERGIA E SUSTENTABILIDADE

CAPÍTULO 7..... 98

GENERACIÓN DE ENERGÍA ELÉCTRICA MEDIANTE MICROTURBINAS HIDRÁULICAS

Nelson Ramiro Gutiérrez Suquillo

Luis Miguel Sánchez Muyulema

Jonnathan Ismael Chamba Cruz

Guillermo Raúl Tumalli Naranjo

 https://doi.org/10.37572/EdArt_2809263077

CAPÍTULO 8..... 114

ENERGÍA DE LAS MAREAS, AGUA Y AIRE

Antonio Rabina Merelas

 https://doi.org/10.37572/EdArt_2809263078

CAPÍTULO 9.....128

EXPOSICIÓN A HIDROCARBUROS AROMÁTICOS POLICÍCLICOS POR EL USO DE LEÑA EN LA COMUNIDAD LA MARCELA, MIQUIHUANA, TAMAULIPAS

Lorenzo Heyer Rodríguez

Valeria Navarro López

Bárbara Azucena Hernández Macías

Elizabeth del Carmen Andrade Limas

Jacinto Treviño Carreón

 https://doi.org/10.37572/EdArt_2809263079

CAPÍTULO 10..... 148

TALLER SOBRE FARMACIA Y COCINA VIVIENTE EN LA RESERVA DE LA BIÓSFERA SIERRA DE HUAUTLA

Amanda Ortiz Sánchez

Angélica María Alemán Octaviano

 https://doi.org/10.37572/EdArt_28092630710

CAPÍTULO 11.....159

BEYOND PUBLIC ACCEPTANCE: REFRAMING NUCLEAR ENERGY COMMUNICATION IN INDONESIA

Muhammad Yunus Zulkifli

Yunita Permatasari

Atika Ratna Sari

 https://doi.org/10.37572/EdArt_28092630711

FORMAÇÃO E COMUNICAÇÃO NA ERA DA INTELIGÊNCIA ARTIFICIAL

CAPÍTULO 12171

ANÁLISIS DEL RENDIMIENTO ACADÉMICO MEDIANTE TÉCNICAS ESTADÍSTICAS MULTIVARIANTES: UN ENFOQUE REPRODUCIBLE CON RMARKDOWN

Alberto Hananel Baigorria

 https://doi.org/10.37572/EdArt_28092630712

CAPÍTULO 13 194

EL PODCAST EDUCATIVO COMO DISPOSITIVO DE AUDIO-MEDIACIÓN PARA EL DESARROLLO SOCIOEMOCIONAL DOCENTE: ARQUITECTURA, MODELO ECOS Y CONSIDERACIONES ÉTICAS PARA EL DISEÑO DE APPS DE PODCAST SEL

Paula Correa-Gutiérrez

Fabiola Sáez-Delgado

Pilar Jara-Coatt

Natalia Zañartu-Canihuante

Felipe Albarrán-Torres

 https://doi.org/10.37572/EdArt_28092630713

CAPÍTULO 14 217

INNOVACIÓN TECNOLÓGICA EN LA GESTIÓN PÚBLICA: INTELIGENCIA ARTIFICIAL Y CALIDAD DEL SERVICIO AL CIUDADANO

José Ángel Meneses Jiménez

Manuel Rocha Gonzales

Raul Hernando Aguado Apolaya

 https://doi.org/10.37572/EdArt_28092630714

CAPÍTULO 15 230

DIFICULTADES EN LA RESOLUCIÓN DE EJERCICIOS Y HABILIDADES MATEMÁTICAS DE ESTUDIANTES EN LA MATERIA DE LENGUAJES Y AUTÓMATAS I

María Eugenia Carreón Romero

Emmanuel Vázquez Benito

Roberto Carlos Carreón Romero

 https://doi.org/10.37572/EdArt_28092630715

CAPÍTULO 16 243

ANÁLISIS MULTIVARIANTE DEL RENDIMIENTO ACADÉMICO MEDIANTE R: UN ENFOQUE PRÁCTICO Y REPRODUCIBLE

Alberto Hananel Baigorria

 https://doi.org/10.37572/EdArt_28092630716

CAPÍTULO 17268

COMPUTACIÓN AFECTIVA Y DISEÑO TECNOPEDAGÓGICO DE SISTEMAS DE MUNDO VIRTUAL PARA EL ENTRENAMIENTO SOCIOEMOCIONAL DOCENTE: REQUERIMIENTOS, COMPONENTES, PARÁMETROS DE INMERSIÓN Y EL MODELO SEED DE DESARROLLO PSICOEMOCIONAL SIMULADO

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Paulo César Coronado-Sánchez

Paula Correa-Gutiérrez

 https://doi.org/10.37572/EdArt_28092630717

SOBRE O ORGANIZADOR.....295

ÍNDICE REMISSIVO296

CAPÍTULO 5

ROLE OF THE INTERACTION AMYLOSE-NANOCELLULOSE ON THE RHEOLOGICAL PROPERTIES AND MOISTURE SORPTION OF MAIZE STARCH

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on the rheological properties and moisture sorption of maize starch. Native maize starch and waxy-maize starch were used to assess the role of amylose, with nanocellulose incorporated at 0, 2, and 6% (dry basis). Pasting properties were evaluated by Rapid Visco Analysis (RVA), while the evolution of viscoelastic properties during storage was analyzed through dynamic time-sweep rheology. Moisture sorption isotherms were determined by Dynamic Vapor Sorption (DVS) and fitted using the GAB model. In addition, Fourier Transform Infrared Spectroscopy (FTIR) was used to evaluate short-range molecular organization. CNC decreased final and setback viscosities, whereas BC increased both parameters, with the effects being more pronounced in maize starch. Both nanocellulose types modified the kinetics of gel formation and generally promoted less elastic or less ordered gel structures, suggesting interference with amylose self-association and self-assembly. CNC and BC also reduced moisture sorption in maize starch, while limited effects were observed in waxy-maize starch. FTIR results further supported changes in short-range organization and water interactions. Overall, the findings suggest that nanocellulose preferentially interacts with amylose, modifying starch molecular organization and consequently its rheological and moisture sorption properties.

KEYWORDS: amylose; starch; cellulose nanocrystals; bacterial cellulose.

ABSTRACT: This study investigated the interaction between amylose and two types of nanocellulose, cellulose nanocrystals (CNC) and bacterial cellulose (BC), and its effect

ROL DE LA INTERACCION AMILOSA-NANOCELULOSA EN LAS PROPIEDADES REOLOGICAS Y SORCION DE HUMEDAD DE ALMIDON DE MAIZ

RESUMEN: Se estudió la interacción entre la amilosa y dos tipos de nanocelulosa (nanocristales de celulosa CNC y la celulosa bacteriana BC), así como su efecto sobre las propiedades reológicas y sorción de humedad en almidón de maíz. Se utilizaron almidón de maíz nativo y almidón de maíz waxy, incorporando nanocelulosa en concentraciones del 0, 2 y 6 % (base seca). Las propiedades de *pasting* se evaluaron mediante *Rapid-Visco-Analysis* (RVA), mientras que la evolución de las propiedades viscoelásticas durante el almacenamiento se analizó mediante ensayos de reología de barrido de tiempo. Las isotermas de sorción de humedad se determinaron mediante *Dynamic Vapor Sorption* (DVS) y se ajustaron al modelo de GAB. Además, se utilizó espectroscopia infrarroja por transformada de Fourier (FTIR) para evaluar la organización molecular de rango corto. CNC redujo los valores de viscosidad final y *setback* de viscosidad, mientras que BC aumentó ambos parámetros, siendo los efectos más pronunciados en el almidón de maíz. Ambos tipos de nanocelulosa modificaron la cinética de formación del gel y, en general, favorecieron estructuras de gel menos elásticas o menos ordenadas, lo que sugiere una interferencia en la auto-asociación y el auto-ensamblaje de la amilosa. CNC y BC también redujeron la sorción de humedad en el almidón de maíz, mientras que se observaron efectos limitados en el almidón de maíz waxy. Los resultados de FTIR mostraron cambios en la organización de rango corto y en la interacción con el agua. Por lo tanto, los resultados sugieren que la nanocelulosa interactúa preferentemente con la amilosa, modificando la organización molecular del almidón y, en consecuencia, sus propiedades reológicas y de sorción de humedad.

PALABRAS CLAVE: amilosa; almidón; nanocristales de celulosa; celulosa bacteriana.

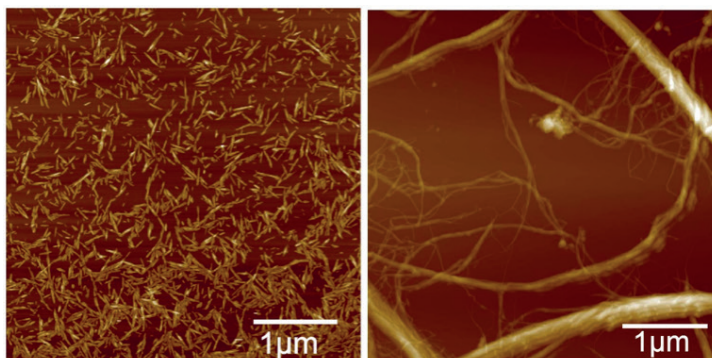
1. INTRODUCTION

Nanocellulose refers to all cellulose-based nanomaterials whose defining characteristic is that at least one of their dimensions (length, width or height) is less than 100 nm. This includes cellulose nanocrystals (CNC), cellulose nanofibers (CNF) and bacterial cellulose (BC). Nanocellulose has been extensively studied in recent decades, primarily due to its potential to reinforce a variety of natural and synthetic materials mechanically (Eichhorn et al, 2022). Furthermore, its plant-based origin makes it interesting from a sustainability perspective, as does its widespread occurrence in nature.

Cellulose nanocrystals (CNCs) are characterized by their rod-like shape, with an average length of 100–200 nm and a width-height of 5–10 nm (Figure 1). They are produced via an acid hydrolysis process, which breaks down the amorphous structures present in elementary cellulose fibers (Fernandes et al, 2023). CNCs also possess a high electronegative charge because of the hydrolysis process. Bacterial cellulose (BC), on the other hand, consists of cellulose fibers produced by the bacterium *Glucacetobacter*

xylinus. This bacterium synthesizes cellulose fibers ranging in length from micrometers, capable of forming entangled networks (Ullah et al, 2016) (Figure 1).

Figure 1. Images captured by Atomic Force Microscopy (AFM) of the nanocellulose used in this study: cellulose nanocrystals (left) and bacterial cellulose (right). Courtesy of Department of Physics, Universidad de Santiago de Chile.



Nanocellulose has a great potential of applications in different fields such as bioengineering, biomedicine, food, health, cosmetics, pharmaceuticals, water treatment, as a structural reinforcement and in the design of new materials, among others (Eichhorn et al, 2022). A relatively new area of technological interest is the development of bio-based materials to be used in the design of multifunctional biocomposites. Although various natural polymers have been tested, starch is one of the most promising alternatives due to its wide availability and low cost, but also for biodegradability and sustainability reasons. Currently, starch is being considering rather than a mere food ingredient. In turn, starch has been proposed for using in different technological applications such as tissue engineering, green packaging, spinning products and in flexible electronic devices, to name a few (Wang et al, 2023).

Using starch in high-tech applications requires a precise understanding of how its structuring polymers (amylose and amylopectin) respond to different processing conditions, as well as how they behave during the storage. Therefore, characterizing the processes of gelatinization and retrogradation is essential for predicting starch performance in various technological applications. In this regard, amylose behavior is significant as it determines the formation of organized structures through self-association and self-assembly mechanisms. Therefore, understanding the interaction amylose-nanocellulose and how the presence of nanocellulose would modify the self-association and self-assembly processes of amylose during the early stages of retrogradation (also known as short-range retrogradation) is important for the rational design of multifunctional starch-nanocellulose composites.

This study aimed to conduct a preliminary analysis of the interaction between two types of nanocellulose (CNC and BC) and amylose in maize starch, and to examine how this interaction affects the rheological properties and moisture sorption of maize starch. A sample of waxy-maize starch was used as a control, and short-range organization analysis carried out by infrared spectroscopy was also tested.

2. PREPARATION OF STARCH-NANOCELLULOSE SUSPENSIONS

Native waxy-maize and maize starch were purchased in powder form from Sigma Aldrich (Germany). Cellulose nanocrystals (CNC) from cotton cellulose pulp were purchased in freeze-dried form from The Process Development Center of the University of Maine (USA). Dried sheets of bacterial cellulose (BC) were kindly provided by Vuelo Pharma (Brazil). AFM images of nanocellulose used in this study are shown in Figure 1. Nanocellulose suspensions were prepared at concentrations of 0%, 2% and 6% w/w (dry basis) in according with Equation 1. Starch was weighed considering having a final starch concentration of 8% w/v.

Equation 1

$$\text{Nanocellulose concentration } \left(\%, \frac{w}{v} \right) = \left(\frac{\text{Nanocellulose}_{\text{weight}}}{\text{Nanocellulose}_{\text{weight}} + \text{Starch}_{\text{weight}}} \right) * 100$$

Well-defined amounts of CNC and BC were weighed and processed before mixing with starch. BC was homogenized using a high-powered kitchen blender for 20 min and vacuum filtered. Filtered BC was used to obtain a hydrated paste with a well-defined concentration. CNC was added directly to water and stirred until complete dispersion. Once CNC and BC were completely dispersed in water, they were sonicated (5 min, 1200 W) at room temperature to promote complete dispersion of the particles. Once the nanocellulose was dispersed the well-defined amount of starch was added.

3. RHEOLOGICAL PROPERTIES

Pasting properties of starch-nanocellulose pastes were evaluated by Rapid-Visco-Analysis (RVA 4500, Perten, Australia) following the protocol described by Díaz-Calderón et al (2018). Briefly, 2 g of native starch was weighed into aluminum canisters and 25 mL of suspension with different CNC or BC concentrations was transferred. Paste profiles were obtained as follow: holding at 25°C for 2 min, heating between 25-95°C at 13.5°C/min, holding at 95°C for 5 min, cooling to 25°C at 13.5°C/min and holding

at 25°C for 5 min. The analysis was carried out under constant stirring (160 rpm). As this work focuses on the interaction between amylose and nanocellulose, this section will only analyze the final viscosity (FV) and setback viscosity (SV) as pasting parameters.

Our results showed large differences on the pasting profiles between starches, but also within the same starch with different levels of CNC or BC (Figure 2). This is reflected in the pasting properties shown in Table 1, which highlight the differences in pasting parameters between maize starch and waxy-maize starch. Our results show that CNC reduces the FV and SV values of wheat and waxy-maize starches, while BC significantly increases them. Literature typically links FV and SV parameters to the ability of starch to reassociate during cooling/storage in an RVA experiment (Balet et al, 2019). Thus, a higher FV value indicates a greater capacity for reassociation. Furthermore, SV has been described as an indicator of retrogradation in starches. However, relatively recent studies on starch-bacterial cellulose blends have shown that assessing the relative increase in viscosity during the cooling stage does not necessarily mean that higher viscosity indicates greater starch association (Díaz-Calderón et al, 2022; Criquet et al, 2026). In this study, the relative increase was found to be around 1.85 for wheat starch mixtures with CNC-BC and 2.05 for waxy starch mixtures with CNC-BC. Therefore, complementary analysis is required to unravel the interaction amylose-nanocellulose.

Figure 2. Pasting profiles of maize starch and waxy-maize starch in presence of cellulose nanocrystals (CNC) or bacterial cellulose (BC). a) maize starch and CNC, b) maize starch and BC, c) waxy-maize starch and CNC, and d) waxy-maize and BC. Dotted lines represent the processing temperature.

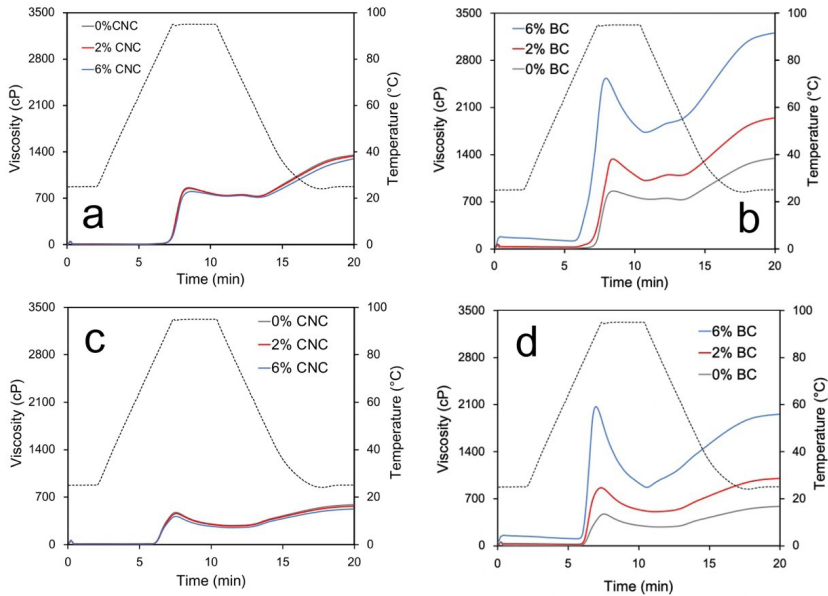


Table 1. Pasting parameters of starch-nanocellulose pastes from the testing by RVA. FV: final viscosity; BV: setback viscosity. Different letters within the same column mean significant differences ($p < 0.05$, $n \geq 3$).

Nanocellulose	Nanocellulose concentration (% db)	Maize		Waxy Maize	
		FV (cP)	SV (cP)	FV (cP)	SV (cP)
CNC	0	1348 ^a	617 ^a	587 ^a	307 ^a
	2	1334 ^a	610 ^a	566 ^b	292 ^a
	6	1293 ^b	582 ^b	525 ^c	276 ^b
BC	2	1994 ^c	928 ^c	1003 ^d	495 ^c
	6	3203 ^d	1473 ^d	1857 ^e	923 ^d

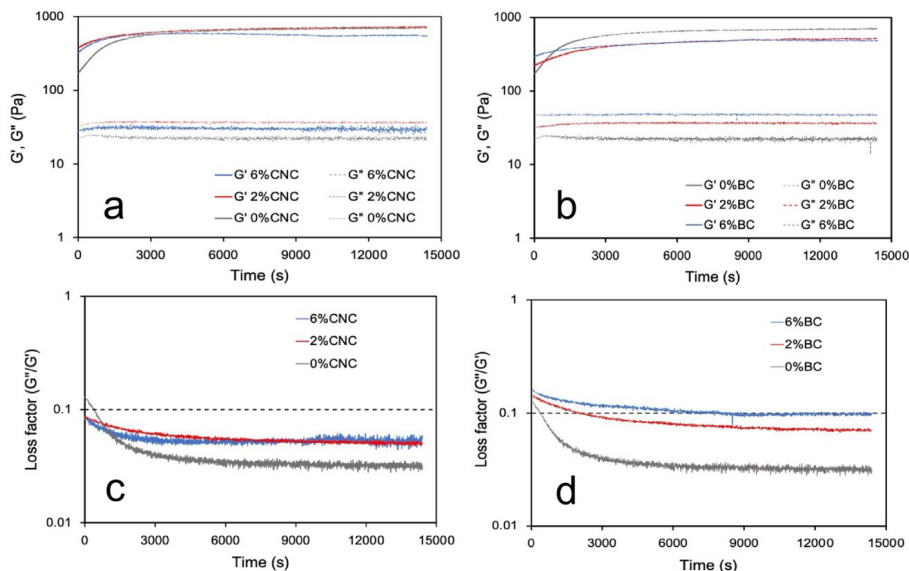
An analysis of dynamic time sweep was performed in a rheometer to assess the change in viscoelasticity over time. A sample was taken from the canister after the RVA test and immediately loaded into a rheometer (HR-2, TA Instruments, USA) set at 25°C. A parallel flat stainless-steel geometry of 50 mm diameter was used, and a 1000 μm gap was selected for testing. G' , G'' and loss factor were determined at constant strain and frequency (0.05% and 10 rad/s respectively) over a period of 4 h at 25°C. Results are presented in Figure 3 and 4.

Following the same trend previously observed in RVA results, changes in G' and G'' over time are quite different between starches and within the same starch with different levels of CNC or BC (Figures 3 and 4).

Our results showed that both starches exhibited “gel-like” behavior in the presence of CNC, with $G' > G''$. However, the presence of CNC accelerated the rate of change in G' in maize starch compared to the control sample (without nanocellulose), as evidenced by the fact that the G' plateau was reached more quickly. Nevertheless, the sample containing 6% CNC exhibited a lower G' value at the end of the analysis (Figure 3a). Furthermore, CNC increased the G'' value throughout the entire time window studied. In the case of waxy-maize starch (Figure 3b), only the sample containing 6% CNC exhibited an increase in G' , a trend also observed in the G'' kinetics. Interesting is the behavior exhibited by the loss factor (G''/G') in starches in the presence of CNC. Our results showed that, in maize starch, the presence of CNC tends to increase the loss factor value, regardless of its concentration (Figure 3c). While all maize-CNC gels can be classified as strong gels according to Ikeda et al (2001) classification (loss factor > 0.1), gels containing CNC tend to be less elastic, suggesting different, possibly less ordered, structural organization. Conversely, the presence of CNC in waxy maize starch does not alter the behavior of the loss factor, which tends to remain at around 0.1 in all cases (Figure 3d). This suggests that CNC interacts preferentially with amylose rather than amylopectin in starches, presumably

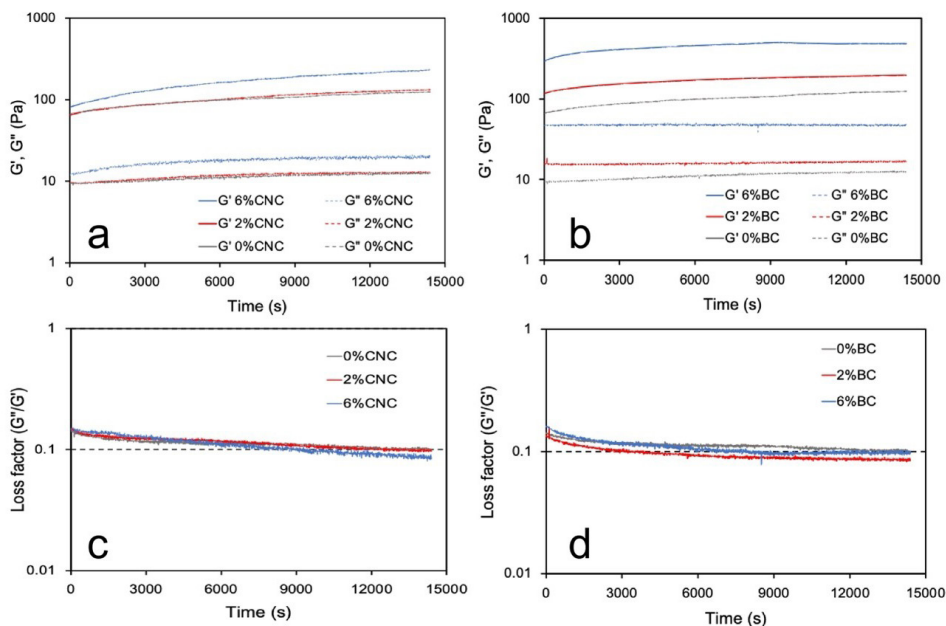
altering the mechanism of self-association of the amylose resulting in less ordered organization and less elastic gels.

Figure 3. Dynamic time sweep of maize starch in presence of cellulose nanocrystals (CNC) or bacterial cellulose (BC). Maize starch and CNC (a and c). Maize starch and BC (b and d). All analysis performed at 25°C, strain of 0.05% and frequency of 10 rad/s.



In the case of BC, the kinetics of the change in G' over time exhibit a distinct pattern. For maize starch, the presence of BC was found to alter the kinetics, resulting in lower G' values compared to the control sample after four hours of analysis (Figure 4a). G'' , on the other hand, increased proportionally to the BC concentration throughout the entire analysis period. In waxy maize starch, the presence of BC produced a significant increase in the rate of change of G' and G'' proportional to the BC concentration (Figure 4b). Analysis of the loss factor (G''/G') showed that BC significantly increased its value, resulting in less elastic gels. In the case of 6% BC, this favored the formation of a weak gel (loss factor >0.1). As in the case of CNC, the presence of BC did not significantly alter the loss factor parameter value in waxy maize starch, which was around 0.1 in all cases. These results are consistent with previous observations in the starch-CNC system and support the idea that, regardless of size and charge, nanocellulose tends to interact preferentially with amylose. This would interfere with the self-association mechanism and favors the formation of weak or less ordered gels.

Figure 4. Dynamic time sweep of waxy-maize starch in presence of cellulose nanocrystals (CNC) or bacterial cellulose (BC). Waxy-maize starch and CNC (a and c). Waxy-maize starch and BC (b and d). All analysis performed at 25°C, strain of 0.05% and frequency of 10 rad/s.



A recent study by Díaz-Calderón et al. (2026) demonstrated how CNC and BC modify the rheological properties of wheat starch, showing that CNC promotes the formation of strong gels by forming aggregates with starch. In contrast, BC promotes the formation of weak gels, as the organization of the gel's structure is characterized by structural domains rich in starch and others rich in BC. The complex structure of BC does not allow for close interaction with other materials (Quero et al., 2015; Díaz-Calderón et al., 2018). Interaction between amylose and biopolymers resulting in less elastic structures due to disruption of amylose helical formation has been previously reported in the literature (Montes et al., 2022; Liu et al., 2021).

4. MOISTURE SORPTION ISOTHERMS

Figure 5. Sorption isotherms of maize starch (a and b) and waxy-maize starch (c and d) in presence of cellulose nanocrystals (CNC) and bacterial cellulose (BC) at different concentrations (0 to 6% db), at 20°C. Continuous lines represent the GAB fit of experimental data.

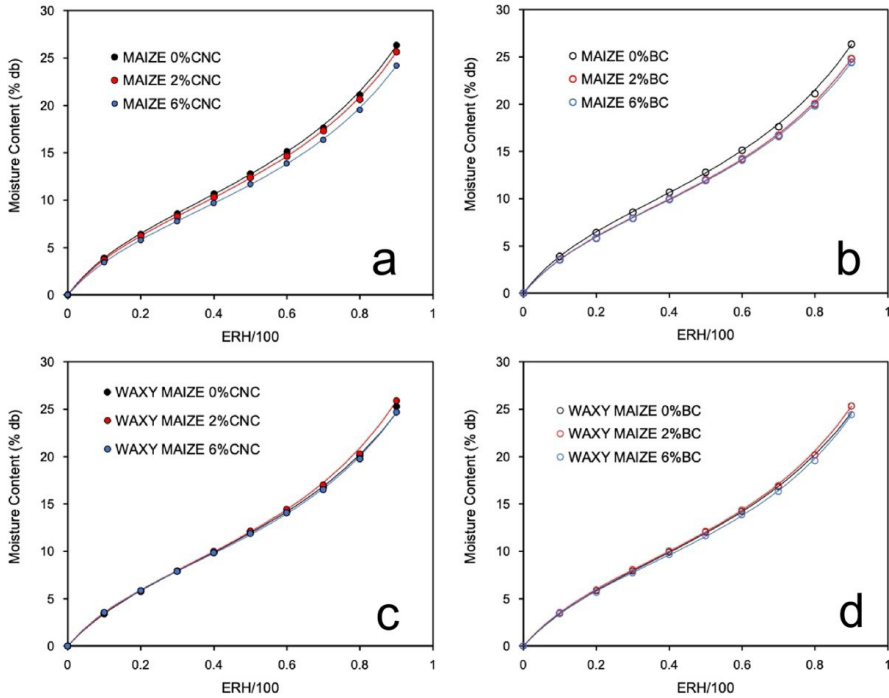


Table 2. Sorption parameters obtained from the GAB fitting.

Starch	Nanocellulose	Concentration (% db)	m_0 (%db)	C-GAB (dimensionless)	K-GAB (dimensionless)	MSE (%)
Maize	CNC	0	10.39	7.05	0.71	0.64
		2	10.09	6.83	0.71	0.33
		6	9.69	6.52	0.70	0.48
	BC	2	9.91	6.63	0.70	0.69
		6	9.88	6.62	0.70	0.53
Waxy Maize	CNC	0	10.26	6.02	0.69	0.45
		2	9.96	6.17	0.72	1.00
		6	9.55	6.86	0.71	0.66
	BC	2	10.00	6.42	0.71	0.57
		6	9.45	6.67	0.71	0.62

An interesting approach for testing structural changes in biopolymers is analyzing the interaction water-matrix. Any change in the association or assembly of the polymer

chains could provoke changes in the moisture sorption behavior. Hence, moisture sorption isotherms of blends starch-nanocellulose were obtained by Dynamic Vapor Sorption (Intrinsic DVS, Surface Measurements Systems, USA). Approximately 20 mg of sample was loaded into the instrument and programmed cycle of equilibrating relative humidities went from 0 to 90%, with 10% increments at constant temperature (25°C). Helped by a microbalance, the samples were at equilibrium when the slope of the change in mass with time (dm/dt) was set to be less than 0.001 mass%/min. The moisture content of the equilibrated sample was expressed in dry-mass basis. The mass of the dry sample was obtained from the first step of the protocol in which the sample was held at an RH of 0% during at least 12 h at 25°C. Once the isotherm is obtained, it was fit by the well-known GAB (Guggenheim-Anderson-de Boer) model.

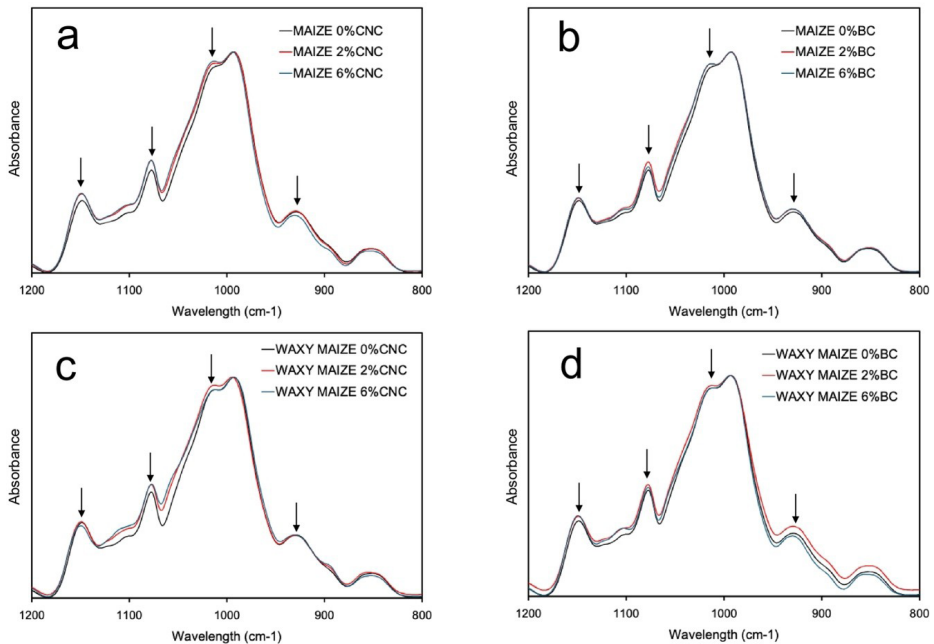
Sorption isotherms of maize starch and waxy-maize starch in presence of CNC and BC are shown in Figure 5. Our results clearly demonstrate that the presence of nanocellulose alters moisture sorption, with this effect depending on the type of starch. Thus, both CNC and BC reduce moisture sorption in maize starch (Figures 5a and 5b). This behavior could be explained by the interaction between amylose and nanocellulose, which probably occurs at the polar sites that were previously available for water. This suggests that changes occur in the self-association and self-assembly of amylose in the presence of nanocellulose during starch retrogradation. This behavior is reflected in the GAB parameters, which show a reduction in moisture content (m_0) and sorption energy (C-GAB parameter) in samples containing nanocellulose (Table 2). Therefore, CNC and BC would occupy polar sites, reducing their availability for water molecules and resulting in weaker binding of water molecules to polar sites. In the case of waxy-maize starch, the presence of nanocellulose does not affect moisture sorption (Figure 5, c and d). This finding supports the idea that nanocellulose primarily interacts with amylose, which in turn interacts with water. Additionally, the C-GAB tends to increase with increasing nanocellulose concentration (Table 2), suggesting that the absence of amylose determines different amylopectin-nanocellulose assemblies.

The literature reports that nanocellulose generally reduces the sorption of moisture in starch matrices, whether in the form of CNC or cellulose nanofibers. This effect has been attributed to greater tortuosity of the water diffusion path, increased crystallinity of the matrix, and interactions between starch and nanocellulose (Popescu et al, 2017; Lin Ong et al, 2020; Lai et al, 2023). Some studies have also reported decreases in GAB model parameters, particularly m_0 , although the effect on C-GAB may vary depending on the type of starch and nanocellulose used (Balakrishnan et al, 2018; García-Guzmán

et al, 2024). Overall, the results suggest that nanocellulose modifies the structure and the interactions between water and starch, thereby reducing the material's moisture absorption capacity.

5. SHORT-RANGE ORGANIZATION BY FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

Figure 6. FTIR patterns of maize starch (a and b) and waxy-maize starch (c and d) in presence of cellulose nanocrystals (CNC) and bacterial cellulose (BC) at different concentrations (0 to 6% db Arrows indicate peaks located at 1150cm⁻¹, 1080cm⁻¹, 1022cm⁻¹ y 930cm⁻¹.



To understand the potential role of molecular arrangements on some macrostructural features an analysis of the short-range organization can be carried out. The short-range organization of blends starch-nanocellulose was studied by Fourier Transform Infrared Spectroscopy (FTIR). Spectra were recorded using a Spectrum Two spectrometer (Perkin Elmer, USA) equipped with an attenuated total reflectance (ATR) accessory. The averaged spectra were obtained over a wavenumber range of 4000 to 400 cm⁻¹ acquiring 64 scans at a spectral resolution of 4 cm⁻¹. All measurements were carried out in triplicate. The spectra obtained were processed using Spectrum 10TM software including baseline correction and normalization of the relative absorbance.

Figure 6 shows the FTIR spectra of maize starch and waxy maize starch mixtures with different concentrations of CNC and BC. Peaks at 1150 cm^{-1} (ordered structures), 1080 cm^{-1} (crystallinity), 1022 cm^{-1} (amorphous structure) and 930 cm^{-1} (water interaction) are of interest when evaluating the short-range organization of the starch (Warren et al., 2016; Falade et al., 2022; Lu et al., 2021). Our results suggest that the presence of nanocellulose alters the short-range organization of the starch. In maize starch, CNC increases the intensity of the peak associated with amorphism (1022 cm^{-1}), suggesting it promotes changes to the starch's self-association and self-assembly mechanisms during storage. This higher degree of starch amorphism could explain the less elastic gel state caused by CNC, which was previously observed in the time-sweep analysis (Figure 3). Furthermore, the peak associated with water interaction (930 cm^{-1}) shows that the peak intensity is slightly lower in samples containing nanocellulose. This is consistent with the behavior of moisture sorption (Figure 2a) and the GAB fit parameters (Table 2). These results are also consistent with the amylose–CNC interaction effect described in the literature. This effect would explain the reduction in sorption (the CNC would interact with the amylose at polar sites where water previously interacted) and the modification of maize starch rheology. Conversely, the increase in peaks associated with organization or crystallinity (1150 cm^{-1} and 1080 cm^{-1}) is interesting; however, this may be more closely related to nanocellulose's contribution to crystallinity/organization than to an effect on the starch structure. In the case of maize starch-BC, the presence of BC results in increased amorphism (1022 cm^{-1}), a slight increase in the water interaction peak (930 cm^{-1}), and minor increases in the crystallinity/organization peaks (1150 cm^{-1} and 1080 cm^{-1}). These results suggest that the mechanisms of amylose–BC interaction differ from those previously observed with CNC. BC would promote short-range interactions with water, but these would not be sufficient to alter moisture sorption on a macro scale. Regarding waxy-maize starch, only 2% of CNC or BC slightly increased the amorphism of starch (Figure 6c). The presence of CNC didn't alter the interaction peak with water (930 cm^{-1}), which is consistent with the sorption behavior, whereas BC changed the water peak intensity following a similar trend presented by the sorption data (Figure 5d). Also, nanocellulose slightly increased the peaks related with organization/crystallinity. Finally, FTIR data supports the idea that nanocellulose interacts preferably with amylose, suggesting that interactions taking place at the molecular level have direct effect on macrostructural properties. Thus, our results showed that the interaction between amylose and nanocellulose is a key mechanism that changes the molecular organization of maize starch, and therefore its rheological and moisture sorption properties.

6. ACKNOWLEDGEMENTS

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ÍNDICE REMISSIVO

A

Agentes conversacionais basados en LLM 269

Agua 59, 98, 101, 103, 104, 107, 108, 109, 110, 111, 112, 114, 116, 117, 118, 119, 120, 121, 122, 123, 124, 126, 127

Agua potable 98, 101, 103, 107, 108, 110, 111, 112

Aire 114, 115, 116, 117, 118, 119, 120, 121, 126, 127, 128, 129, 130, 131, 132, 134, 136, 138, 139, 140, 141, 142, 143, 144

Aire interior 128, 129, 130, 131, 132, 136, 138, 139, 140, 141, 142, 143, 144

Algoritmo computacional 2, 7, 14

Amylose 58, 60, 61, 62, 63, 64, 65, 67, 69

Análisis multivariante 171, 191, 243, 258, 267

Analítica multimodal del aprendizaje 268, 269, 270, 272, 281, 288

Arquitectura de software 269, 289

Audio-mediación 194, 195, 196, 197, 198, 203, 208, 213

Autómatas 230, 231, 233, 234, 235, 236, 237, 241

B

Bacterial cellulose 58, 59, 60, 61, 62, 64, 65, 66, 68, 70, 71

Benzo(a)pireno 128, 129, 136, 143

Bienestar docente 195, 198, 208, 214, 289, 291

Biorretroalimentación 268, 269, 283, 284, 285, 286, 288

C

Calidad del servicio 217, 218, 219, 220, 221, 222, 223, 224, 225, 226, 227, 228, 229

Caras de Chernoff 171, 175, 178, 186, 187, 190, 191, 243, 245, 262, 263, 264, 266

Cellulose nanocrystals 58, 59, 60, 61, 62, 64, 65, 66, 68, 71

Cizallamiento 32, 33, 34, 35, 36, 37, 38, 39, 40, 44, 45

Competencias socioemocionales docentes 195, 202, 213

Computación afectiva 268, 269, 270, 273, 274, 289

Conocimiento local 148

Contaminación 129, 130

D

Desarrollo sostenible 98, 113

Dificultades 56, 91, 230, 232, 233, 234, 241, 242

E

Educación en ingeniería 72, 76, 77, 81, 242

Energía hidroeléctrica 98

Energía maremotriz 114

Energía Renovable 98, 113

Entrenamiento Socioemocional Docente 268, 269, 271, 289

ERIM 46, 47, 48, 50, 51, 52, 55, 56

Escritura académica 72, 74, 76

Estadística aplicada 243

Ética del diseño tecnológico 195

F

Fiabilidad 46, 47, 50, 55, 114, 217, 220, 222, 223, 225, 226, 227, 228

Fidelidad psicológica 269, 270, 280, 283, 289

Formación investigativa 72, 74, 76, 77, 79, 96

Formulación lagrangiana 2, 4

Frequency analysis 16

Fuerza de corte 32, 33, 35, 36, 39, 41, 42, 44, 45

G

Gestión pública local 217, 219, 226

H

Huertos familiares 148, 149, 150, 152, 154, 157

I

Inteligencia artificial 72, 74, 75, 76, 77, 78, 81, 88, 89, 96, 197, 211, 212, 214, 217, 218, 219, 222, 223, 224, 225, 226, 227, 228, 229, 271, 272, 288

Inteligencia artificial generativa 72, 74, 76, 77, 89, 96, 272

K

Knowledge translation 160, 164, 165, 166

M

Marco teórico 72, 73, 74, 75, 76, 77, 78, 79, 80, 81, 87, 88, 89, 91, 92, 93, 94, 95, 96, 97, 202
Mareas 114, 115, 116, 117, 118, 122, 123, 126
Matemáticas 230, 231, 232, 233, 234, 235, 236, 237, 240, 241, 242
Mechanical systems 16
Media representation 160, 161, 163
Microturbina 98, 108, 109
Modelo dinámico 1, 2, 3, 4, 7, 14
Modelo ECOS 194, 195, 198, 204, 205, 206, 207, 209, 210, 211, 213
Modelo SEED 268, 269, 270, 284, 285, 286, 289, 290
Modelo SERVQUAL 217, 220, 222, 224
Mundo virtual 268, 269, 270, 271, 284, 288, 289

N

Non-destructive testing 16, 20
Nuclear energy communication 159, 160, 161, 164, 166, 167, 168, 169

P

Plantas alimenticias 148, 149, 151
Plantas medicinales 148, 149, 150, 151, 154, 158
Podcast educativo 194, 195, 196, 197, 198, 213
Predictive maintenance 16
Public meaning 160, 161, 163, 167, 168

R

Razonamiento estadístico 171, 173, 175, 176
Realidad virtual inmersiva 269
Rendimiento académico 171, 178, 180, 243, 257, 259, 261, 266
Reproducibilidad 171, 172, 173, 174, 175, 176, 188, 190, 191, 243
Resolución ejercicios 230
Resonancia 114, 115, 122, 123, 124, 126, 127
RMarkdown 171, 172, 175, 176, 179, 188, 189, 191

S

Science communication 160, 162, 163, 167, 168, 169, 170
Silueta 32, 185

Starch 58, 60, 61, 62, 63, 64, 65, 66, 67, 68, 69, 70, 71

Structural health monitoring 16

T

TPM 46, 47, 50, 51, 57

Transformación digital 217, 219, 221

V

Vibration-based identification 16

Vibration diagnostics 16, 18, 20

