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Relevance of Scanning Electron Microscope and Nuclear Magnetic Resonance Technique in Food Research: A Review

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ABSTRACT

The aim of this review was to investigate the relevance of scanning electron microscope (SEM) and nuclear magnetic resonance (NMR) in research in the food industry. The microstructure of food is affected by food processing. The food components such as proteins, vitamins and starch are usually involved in irreversible changes during processing. The proteins are denatured most times while the water soluble vitamins are lost. A food processor needs to ensure that quality control is put into consideration during production process. Globalization has also made the world a global village whereby processed food products produced in one end of the globe finds its way to another end. Different processing methods affect processed food, hence instrumentation techniques such as the use of SEM and NMR comes in handy. The major limiting factor in their use and installation is cost because they are expensive equipment but have high level of accuracy. SEM and NMR are nondestructive analytical technique. The two Technology have been adopted in the investigation of microstructure of food. SEM has been used to view Salmonella biofilm, interaction between some fish products and the inner part of the packaging material. NMR instrumentation was first used for moisture measurement in foods.

Keywords: Microstructure, nondestructive, instrumentation, Quality control

INTRODUCTION

A scanning electron microscope is an instrument that produces images of a sample by scanning the surface of the sample using a focused beam of high-energy electrons. These high-energy electrons interact with the atoms in the sample, and produces various signals containing information about the surface topography and composition of the samples. The position of the beam combines with the intensity of the detected signal to produce an image (Schmitt, 2014). The first 'scanning microscope' was built in 1935 by a German scientist, Max Knoll. The major limiting factor in the use and installation of SEM is the cost because it is an expensive equipment but has a high level of accuracy. SEM Techniques promises to be very useful in food research. SEM has enabled the food industry to broaden possible analysis to which samples can be subjected to and this has improved quality control techniques. Different food processing conditions affect the microstructure of food and this can be investigated using SEM. SEM is used to identify the structure of food which includes the intercellular spaces (Jarzębski *et al*., 2017; Karim *et al*., 2018). SEM can play a significant role in investigating the microstructure of agricultural products during drying or cooking as this affects the behaviour of the product, for example, a product with a porous microstructure will behave differently from one with a compact microstructure and this goes a long way in providing information on the nature of processing to be given to such a product (Xiao and Gao, 2012). Live and dead spores of microbial cells, food spoilage organisms and the extent of thermal damage to these cells could be viewed using SEM (Rozali *et al*., 2017). SEM can be used to investigate disease-causing microbial cells on food samples (Gatti and Montanari, 2018). Ultrastructural information is provided by electron microscopy (Kanemaru *et al*., 2009). Surface geometry of food powders can be investigated using SEM (Burgain *et al*., 2017). Based on literature, SEM has been extensively used in food research.

Newer and easier methods of analytical determinations in research are constantly in investigation in the food industry so as to ensure better quality of processed food products. NMR and SEM are becoming popular in investigations regarding the microstructure of foods. Investigation of processed food microstructure is important as it helps in determining its functional properties such as rheology and water holding capacity (Wang *et al*., 2018). Kaláb *et al.* (1995) reported that food microstructure evaluation is most appropriate with microscopy and imaging techniques because images are formed which can be converted to numerical data if there is a need. Some other techniques used in different investigations in the food industry include X-ray microtomography (Tan *et al*., 2016), fourier transform infrared spectroscopy (FTIR) (Jiao *et al*., 2018), SDS-PAGE (sodium dodecyl sulfate polyacrylamide gel electrophoresis) (Liu *et al*., 2019), mercury intrusion porosimetry (Kassama and Ngadi, 2005), differential scanning calorimetry/calorimeter (DSC) (Farah *et al*., 2018), X-ray diffractometry/diffractometer (XRD) (Mondal *et al*., 2015), atomic force microscopy (AFM) (Shi *et al*., 2018), Laser scanning confocal microscopy (LSCM) (Corradini and McClements, 2017). The properties and technological benefits of nanomaterials has necessitated its inclusion in food products, consequently, appropriate analytical techniques are required to characterize these nanomaterials (Busquets, 2017). Laghi *et al*. (2014) classified NMR technique as a powerful technique for food researchers and reported that NMR can be used to investigate the structure of any molecule which makes it a good tool in the food industry. NMR is an important tool in addressing food adulteration in the case where legal substances have been mixed with unsafe substances because NMR can measure isotopic ratios directly at different positions in a given molecule therefore NMR is helpful in food authentication (Ko and Hsieh, 2018). Foods from different origin have been subjected to food authentication testing such as tomatoes, vinegar and milk (Sobolev *et al*., 2016). Detecting complex samples in food accurately and promptly is necessary for human health safety (Gu *et al*., 2018). NMR can be used to determine the dynamics of protein (Wu *et al*., 2019). Borthakur *et al*. (2016) reported that nanoemulsions used in food industries are characterized using NMR. Verboven *et al*. (2018) defined food microstructure as the arrangement and interaction in food constituents which results in a microscopic matrix. Textural and structural changes usually lead to changes in the innate properties of food hence the need to investigate the structure of processed food. Processing methods requiring high temperature such as extrusion cooking (Fellows, 2017) and deep-fat frying (Kerr, 2016) could transform proteins and polysaccharides in foods. Extrusion cooking is a technology which involves high temperature in most cases and would likely result in microstructural changes. Most researchers have investigated the microstructure of foods with the aid of NMR analytical techniques. The aim of this review was to investigate the relevance of NMR and SEM in the food industry mostly in food microstructure determination. Since SEM and NMR has been used in food applications by many researchers, this proves that SEM and NMR are actually important, hence has prompted the objective of this review to investigate the relevance of NMR and SEM in the food industry mostly in food microstructure determination.

Literature for the review was sourced mainly from Science Direct, PubMed and Google and Mendeley Reference Manager was used to insert in-text references and the list of Bibliography.

SEM Applications in food research

Dhowlaghar *et al*. (2018) used SEM to view Salmonella biofilm from catfish mucus formed on several food-contact surfaces. Kontominas *et al*. (2006) used SEM to investigate the interaction between some fish products and the inner part of the packaging material (which is a metal). dos Santos Rodrigues *et al*. (2017) observed the ultrastructure of cells in *Staphylococcus aureus* biofilm. Nowacka *et al*. (2018) investigated the suitability (level of penetration of substances such as moisture) of some packaging materials (paper and cardboard) used in food. Liu *et al*. (2018) determined the different microstructures of milk proteins printed with 3D printer system for food. Liu and Lanier (2015) characterized the microstructure of ground meat. Nguyen *et al*. (2018) analyzed the structure of potato slabs during drying. Ray *et al*. (2015) examined the structure of the shell of table eggs. Mahato *et al*. (2024) determined the quality of eggshells by determining the thickness of the eggshells. Işbilir *et al*. (2025) used light and SEM to examine the intestinal sections in turkeys. The samples for SEM analysis are managed in different ways such as maintaining the samples at low temperature to prevent a change in the morphology and structure of the samples when in vacuum. Also, the samples are coated by films which are impermeable and conductive so as to dehydrate the sample thereby preventing dispersion of water into vacuum. These measures ensure accurate results (Alberici & Kostal, 2015). Rovira *et al*. (2011)

investigated the microstructure of cheese curd from goat milk. Tan and Balasubramanian (2017) investigated the microstructure of cocoa powder during processing. Riaz *et al*. (2018) characterized an active food packaging material. Ayala-Hernandez *et al*. (2008) and Martin *et al*. (2006) observed the relationship between exopolysaccharides and proteins in milk. Hondoh *et al*. (2016) observed the movement of oil into chocolate. Ong *et al*. (2011) investigated the microstructural changes in milk during cheese manufacture. James and Smith (2009) characterized the microstructure of chocolate during the processes of heating and cooling. Jackowiak *et al*. (2005) characterized wheat kernels which have been damaged by *Fusarium*. Barron and Butler (2008) characterized the microstructural properties of bread crumb. Dalgleish *et al*. (2004) observed casein micelles. Zhao *et al*. (2011) characterized powders from soy bean proteins. Taglienti *et al*. (2011) investigated the pulp of kiwifruit. Han *et al*. (2016) investigated *Vibrio parahaemolyticus* biofilm on the surfaces of some food and surfaces in contact with food. Oh *et al*. (2016) used SEM to directly count bacterial cells on the surfaces of treated and untreated disposable gloves used in food handling. James and Yang (2011) investigated the tenderness of beef which has been subjected to different processing treatments by observing the microstructure using SEM. Shrestha *et al*. (2015) quantified the changes in the structure of extruded maize starch during amylase digestion. Xiao *et al*. (2013) investigated the structure of sorghum lignin. Jafari *et al*. (2018) investigated the microstructure of dough and bread. Liu *et al*. (2019) investigated the characteristics of dough produced from damaged cassava starch. García-García *et al*. (2018) investigated water mobility and structure of ham during processing. Philipp *et al*. (2017) investigated microstructural characteristics of extruded snack from rice starch and pea protein. Zhang *et al*. (2018) investigated the microstructure of probiotic containing cereal-based food product. Wang *et al*. (2019) investigated microstructure of buckwheat noodles. Martínez *et al*. (2015) investigated the microstructure of extruded wheat flour. Kharat *et al*. (2019) investigated extrudates from different varieties of whole grain millet flours. Nwadi and Okonkwo (2020) investigated the effect of extrusion cooking on the microstructure of whole wheat flour using scanning electron microscope.

NMR Technique in food research

One of the most detailed and effective analytical technique used to investigate the structure (such as structure of proteins) and composition of food without destroying the sample nor producing harmful substances is (NMR (van Duynhoven, 2017). Callaghan (2017) described NMR microscopy as a Magnetic Resonance Imaging (MRI) technique which is usually conducted in vertical bore NMR magnets which involves small samples. Application of NMR in Food Science started in the 1980s (Marcone *et al*., 2013). In the past NMR was been used only to determine structure but it is now also been used in chemical fingerprinting and study of metabolites (Ramakrishnan and Luthria, 2017). Farag *et al*. (2018) quantified metabolites in two species of cinnamon. NMR is very efficient in identifying and quantifying substances including metabolites within a short time. The content, purity and molecular structure of a sample can be determined using NMR. This technique has been applied in research and as a quality control measure especially in food industries. Chen *et al*. (2025) briefly summarized the recent advances in the use of NMR spectroscopy in phospholipids analysis. Deng *et al*. (2025) comprehensively investigated the purities of four D-amino acids. NMR can be used to analyse all organic compounds (such as amino acid profile) including determination of changes in water distribution and mobility. Heat treatment could have different effects on food, NMR technique can be used to determine these changes. NMR has been applied in determining melamine level in foods. Some foods in which NMR technique has been applied include wheat, vinegar, meat, dairy products (milk and cheese), coffee, green tea, oils (from fish and vegetable), apple juices and more recently wine and beer (Mannina *et al*., 2012). NMR have been used to differentiate soluble and insoluble casein phosphate nanoclusters in milk, investigating the distribution of phosphorus in cheese and detecting the differences in the caseins of different species of milk-producing animals (Boiani *et al*., 2017). The advantages of NMR over other physico chemical techniques is that the sample is not destroyed and there is a wide range of length and time scales from which information is usually acquired, hence the efficiency of NMR in probing food which is a complex system (Belton *et al*., 1993; Watanabe *et al*. 1995). As a result of consumer consciousness regarding the safety as well as quality of food, all the food supply chains are ensuring significant concern for food authenticity. NMR and metabolome field have created great opportunities in the area of food authentication (Lolli & Caligiani, 2024).

Applications of NMR in food research

Srikaeo and Rahman (2018) using NMR obtained valuable data which are helpful in designing rice storage and processing. Gudjónsdóttir *et al*. (2018) using NMR obtained data which is helpful in predicting the general quality characteristics (physicochemical) of Atlantic mackerel. Ohtsuki, Sato, Sugimoto, Akiyama, and Kawamura (2012) used NMR to quantify sorbic acid in food (processed). Carosio *et al*. (2016) measured thermal attributes of oilseeds. Gresley and Peron (2019) identified chocolates from three different geographical regions based on the constituents of the chocolate. Pramai *et al*. (2018) identified metabolites in germinated rice which may have some health and nutritional benefits. Zhao *et al*. (2013) detected *Cronobacter skazakii* in very low concentration using NMR-based assay. Ishihara *et al*. (2018) identified the particular metabolites in cabbage vinegar which differentiates it from other vinegars. Boiani *et al*. (2017) identified different species of phosphorus in skim milk so as to investigate ionic changes during microfiltration and diafiltration. Malongane *et al*. (2018) identified the compounds in four different South African herbal teas. Soares *et al*. (2017) investigated changes (chemical and nutritional) which took place during heat treatment of passion fruit juice. Rodrigues *et al*. (2011) monitored the chemical changes in lager beer under forced aging conditions. Martínez-Yusta and Guillén (2014) determined changes (lipidic composition and Level of degradation) in some fried foods and the frying medium. Rashidinejad *et al*. (2017) investigated interactions between catechins in tea and fat in cheese. Peters *et al*. (2016) determined and characterized the water binding capacity of whey proteins. Cao *et al*. (2015) determined the quantity of sucrose in beverages. Li *et al*. (2018) detected adulteration of peanut oil. Verbi Pereira *et al*. (2015) used NMR combined with chemometric analysis to determine fat content in packaged mayonnaise and salad. Sopelana, Arizabaleta, Ibargoitia, and Guillén (2013) identified and quantified the lipidic composition of margarines. Carneiro *et al*. (2016) investigated the movement of water during salted fish storage. T. Li *et al*. (2015) investigated water dispersal and microstructure of tofu (soybean curd). Otero and Préstamo (2009) reported differences in the content of the main sugars in pressurized and non-pressurized samples of strawberry after an investigation using NMR. Yu *et al*. (2016) investigated the effect of addition of milk solids nonfat on the microstructural and physical properties of yogurts. Park *et al*. (2019) investigated water mobility in turanose-rice flour for noodle-making purposes and reported that two water populations were revealed on NMR relaxation. Zhenbin *et al*. (2018) characterized water mobility and hydrogen bond in mashed potatoes containing different types of gum. Diantom *et al*. (2019) investigated proton molecular mobility in commercial samples of pasta. Manoi and Rizvi (2010) investigated the water solubility of cross-linked starch. Franssen and Boeriu (2014) investigated the level of substitution of the glucose moiety in maize starch esterified with allyl glycidyl ether. Ramesh Yadav, Guha, Tharanathan, and Ramteke (2006) investigated changes in characteristics of processed potato starch. Klaus *et al*. (2015) investigated alkaline and water extracted samples of edible mushroom. Sun *et al*. (2019) investigated oil quality during frying. Proietti *et al*. (2018) investigated moisture profile of pear during drying. Cheng *et al*. (2019) investigated freeze-thaw cycles in beef. Tan *et al*. (2018) investigated freeze-thaw cycles in instant sea cucumber. Xu *et al*. (2016) investigated the characteristics of solid food sample infused with carbondioxide. Kamal *et al*. (2018) investigated microstructure and water dynamics in apple jelly. Traffano-Schiffo *et al*. (2017) investigated dehydrated kiwifruit. Tylewicz *et al*. (2016) investigated the characteristics of freeze-dried and rehydrated apple. Lu *et al*. (2016) investigated the characteristics of intermediate-moisture foods during storage.

CONCLUSION AND FUTURE PERSPECTIVES

SEM and NMR have been extensively used in food research in the laboratory. They are useful analytical techniques which provide more detailed information especially on microstructural arrangement of molecules in a given food sample, they also help in ascertaining the suitability of packaging materials. However, these techniques require practical application during food processing in the food industry. The expensive equipment poses a huge cost constraint for the food processor. Government agencies such as regulatory agencies are expected to play a key role in this area so as to ensure a high quality processed food product for consumers. Since SEM and NMR have been used in food applications by many researchers, this makes it relevant in food research. SEM and NMR techniques promises to be very useful in food research. The major limiting factor in the use and installation of SEM and NMR which is the cost needs to be addressed so as to have a high level of accuracy in food research especially in food industries which will go a long way in addressing quality issues thereby producing safe foods with high quality. Food processors especially at a small scale needs to also make adequate provision for equipment which could be used to ensure adequate quality control of processed food products such as SEM and NMR. In the future, it is expected that in establishing a laboratory for food analyses, every required equipment is supposed to be installed before operations begin. Adequate budgeting should be carried out especially for procurement of expensive equipment such as those for SEM and NMR techniques. Integrating SEM and NMR with newer imaging techniques or AI-based data analysis for improved food authentication may also be considered.

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