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Effect of Using Different Types of Binder on the HA Foam Properties

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Abstract

This study investigates the impact of double sintering on the properties of porous hydroxyapatite (HA) structures, which were fabricated using the replication method. Hydroxyapatite is a widely used biomaterial due to its biocompatibility and similarity to the mineral phase of natural bone. The replication method involves creating porous structures that mimic natural bone tissue. This research aimed to assess the structural and mechanical properties of porous HA and determine whether double sintering can enhance these properties. The double sintering process involves subjecting the porous HA samples to two separate sintering steps, potentially improving densification and crystallinity. Methods included fabricating porous HA samples using the replication method, followed by a single and double sintering process. The samples were characterized using techniques such as X-ray diffraction, scanning electron microscopy, and mechanical testing to assess changes in crystallinity, microstructure, and mechanical strength. Results show that double sintering significantly impacted the properties of the porous HA. It led to improved densification compared to the single sintering process. Additionally, compressive strength was also enhanced in the double-sintered samples. In conclusion, this study demonstrates that double sintering is an effective technique to enhance the properties of porous hydroxyapatite fabricated by the replication method. These improvements in structural and mechanical properties make double sintering a promising method for producing biomaterials with better suitability for bone tissue engineering and other biomedical applications.

1. Introduction

Hydroxyapatite (HA), a ceramic composed of calcium phosphate, has garnered considerable attention in the realms of biomaterials and tissue engineering due to its striking similarity to the mineral composition of natural bone. Its commendable biocompatibility, osteoconductive characteristics, and capacity to facilitate bone tissue growth position it as an ideal candidate for diverse biomedical applications, encompassing bone grafts, implants, and scaffolds. Porous hydroxyapatite materials have demonstrated promise in addressing the demands of bone repair and regeneration. The porous nature of these materials facilitates cell infiltration and the diffusion of bioactive molecules, rendering them suitable for use in tissue engineering. Several fabrication methods have been devised to craft porous hydroxyapatite structures, with the replication method being one of them. This technique involves generating porous structures by replicating a template with the desired morphology.

Although effective in producing intricate geometries, the replication method presents opportunities for refining the properties of resulting porous hydroxyapatite, particularly in terms of mechanical strength and structural integrity. Double sintering, a post-processing technique involving two sintering steps, has been employed in various ceramics to enhance properties by increasing material density and improving crystallinity. However, its application to porous hydroxyapatite created through the replication method remains a relatively unexplored area of research. Consequently, the motivation for this study is to explore the impact of double sintering on the properties of porous hydroxyapatite produced via the replication method. The study seeks to ascertain whether double sintering can bring about improvements in material characteristics, including heightened densification, enhanced crystallinity, and improved mechanical strength, thereby rendering the material more suitable for applications in bone tissue engineering and related biomedical fields.[1]

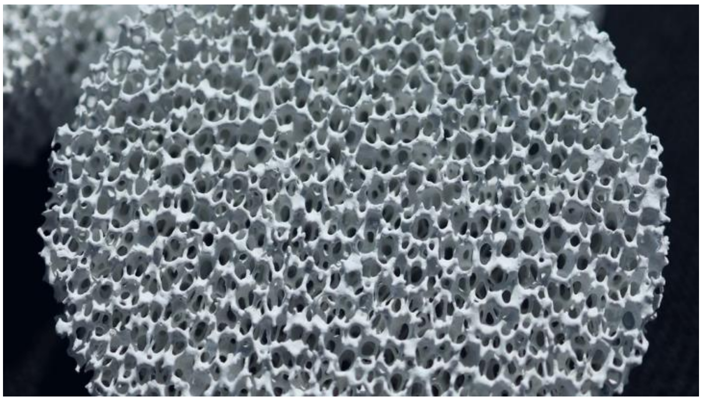


Fig-1 *Porous HA Material [1]*

The term "porous" is used to describe a material or substance that contains pores or tiny apertures through which gases, liquids, or other things can pass. The gaps or empty spaces that exist within a substance are called pores, and they can differ in size and location. When a substance is porous, it signifies that its structure has interwoven voids or spaces in it. This characteristic is frequently preferred in a variety of settings.[2] For instance, the porosity of porous metals or ceramics can be tailored to improve properties like filtration, absorption, or the capacity to support biological processes like tissue growth. Porous HA has been widely used to replace artificial bone. Repair, regeneration, and rebuilding of missing, damaged, or deteriorating tissues are the main goals of tissue engineering. Even though bone tissue has a great capacity for bone regeneration, bone transplants are necessary in cases where there is a large bony defect or when the healing process of the bone is challenging. Matching the osteoconductive characteristics of a porous ceramic scaffold with the osteoinductive or osteogenic characteristics of living bone cells is now vital.[3]

2. Methodology

This process describes a two-step, high-temperature sintering process for creating porous nano-CaP ceramics. The article suggests utilizing sequential freeze-casting as a straightforward and effective approach to fabricating porous HA scaffolds with graded porosity. The polymeric replication sponge method is used to generate porous HA ceramics with different amounts of magnesium. The paper describes a unique dual-phase mixing process for producing porous hydroxyapatite. Regarding the effects of double sintering on the characteristics of porous hydroxyapatite made using the replication process, no information is available. The technique section details the systematic approach employed for synthesising and characterising hydroxyapatite foam, ensuring reliability and consistency in the results. The study design encompasses process parameters, manufacturing techniques, and appropriate precursors. The synthesis process is comprehensively explained, covering methods such as sintering, sol-gel processes, and other advanced techniques for producing HA foam. Significant parameters, such as temperature, pressure, and precursor concentrations, are elucidated to elucidate the controlled formation of the foam structure.

Hydroxyapatite (HA) foam is an essential material for biomedical and technical applications because of its unique properties, which include its osteoconductive Ness, biocompatibility, and similarity to the mineral component of genuine bone. The potential applications of HA foam in bone tissue engineering, drug delivery systems, and scaffolds for regenerative medicine have generated interest as the demand for better biomaterials increases. The search for materials that can fit in perfectly with the biological environment is what has prompted researchers to investigate Research Progress in Mechanical and Manufacturing Engineering, this introduction aims to provide a comprehensive understanding of the significance of HA foam in many areas, including the specific issues that are at hand.

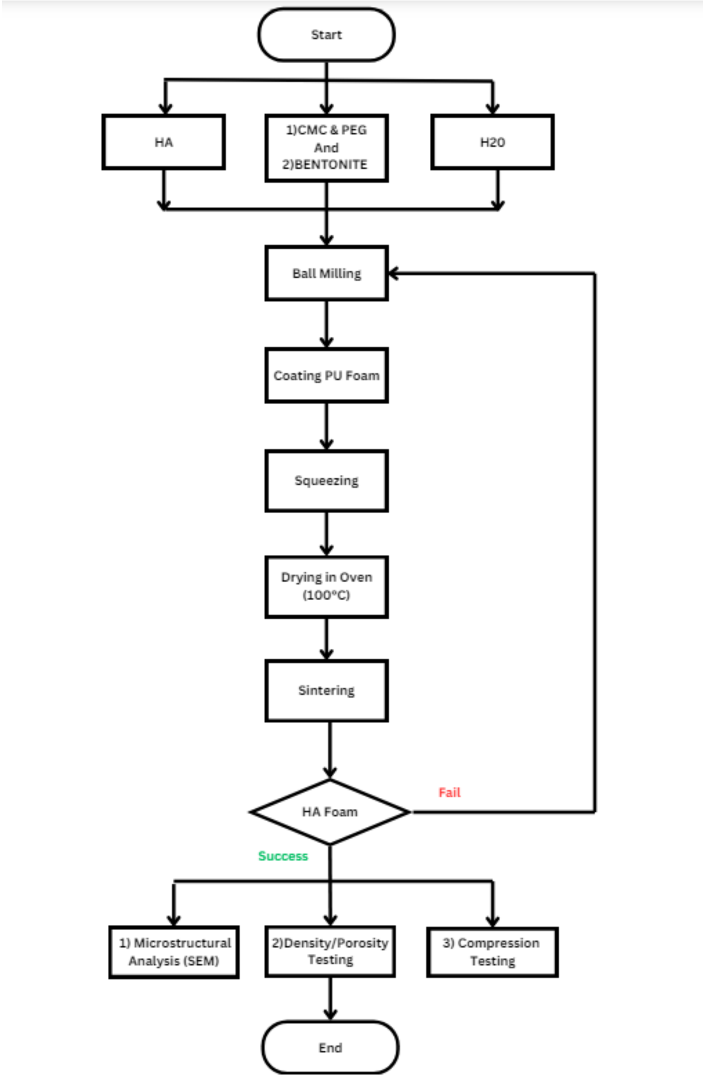


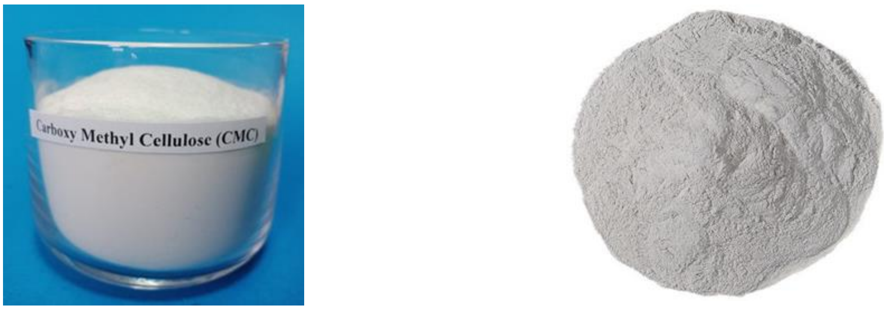
Fig. 1 *Flowchart in sampling and testing*

2.1 Apparatus

Hydroxyapatite powder (Fig. 2a)is the main ingredient used to create the hydroxyapatite foam that is used to make this copy. It is combined with binder powders such as polyethylene glycol (PEG) (Fig. 2b) and carboxymethyl cellulose (CMC) (Fig. 3a). Then, in order to create the slurry at the sponge, all of these ingredients were mixed with distilled water and binder Bentonite (Fig. 3b).

(a) **(b)**

Fig. 2 *(a) Hydroxyapatite (b) PEG*



(a) **(b)**

Fig. 3 *(a) CMC, (b) Bentonite*

2.0 Equipment

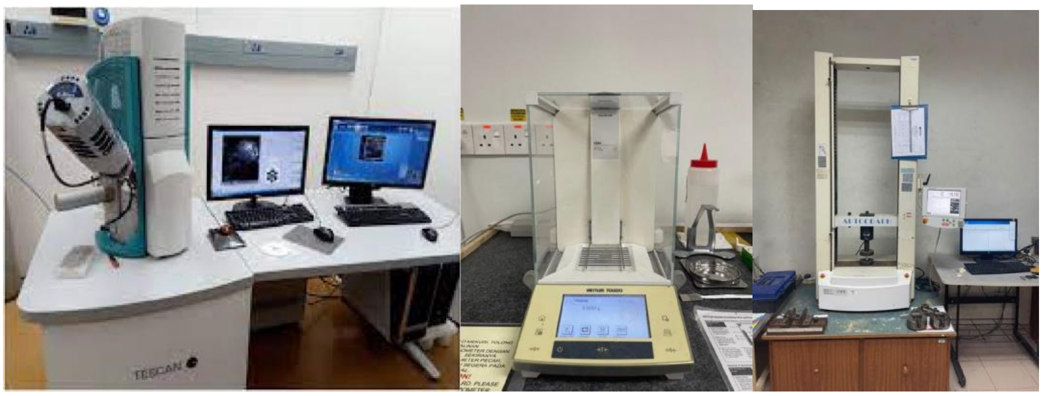
Various tools are utilized to fabricate and analyse hydroxyapatite (HA) foam. These tools include a polyurethane foam template for constructing porous structures, a ball mill or mortar and pestle for grinding raw materials, and sieves for achieving uniform particle size. Using dip-coating machinery, HA slurry is applied to the PU foam, and the foam is then dried in a drying oven. To accomplish densification and crystallization, sintering is done using a sintering furnace for both single and double sintering operations. High Furnace sintered HA characterization entails. Furthermore, tools for measuring compressive strength determine mechanical strength and devices for evaluating porosity and pore dispersion in HA foam are called porosimeters or micro-CT scanners.

(a) **(b)** **(c)**

Fig. 4 *(a) Ball Milling (b) Oven (c) High Furnace*

2.1 Testing Method

Hydroxyapatite (HA) foam is tested using a variety of procedures to evaluate its mechanical and structural characteristics. High-resolution images are obtained by covering the sample with a conductive substance and scanning it with an electron beam in Scanning Electron Microscopy (SEM) (Fig. 5a), which is used to investigate the microstructure and morphology. Fig. 5c show a Universal Testing Machine used for compression tests. By compressing a foam sample until it breaks, mechanical testing—especially compressive strength testing—measures the mechanical strength and load-bearing capability. The compressive strength is then determined using the force and deformation data. The porosimeter (Fig. 5b) evaluates the porosity and pore size distribution by measuring the pore volume and size distribution using methods like gas adsorption or mercury intrusion porosimeter. The information obtained from these techniques is crucial for assessing and improving hydroxyapatite foams in biological applications.



(a) **(b)** **(c)**

Fig. 5 *(a) SEM (b) Porosity and Density (c) Compression Testing*

Apparent porosity is a specific measure of the porosity of a material, typically used in the context of porous ceramics and similar materials. It considers the volume of open pores that are accessible from the surface, rather than including any closed internal pores that are not connected to the surface. The formula for calculating apparent porosity.

$$\text{Apparent Porosity (\%)} = \frac{w_3 - w_1}{w_3 - w_2} \times 100 \quad (1)$$

3. Result and Discussion

3.1 Properties HA Foam binder CMC and PEG

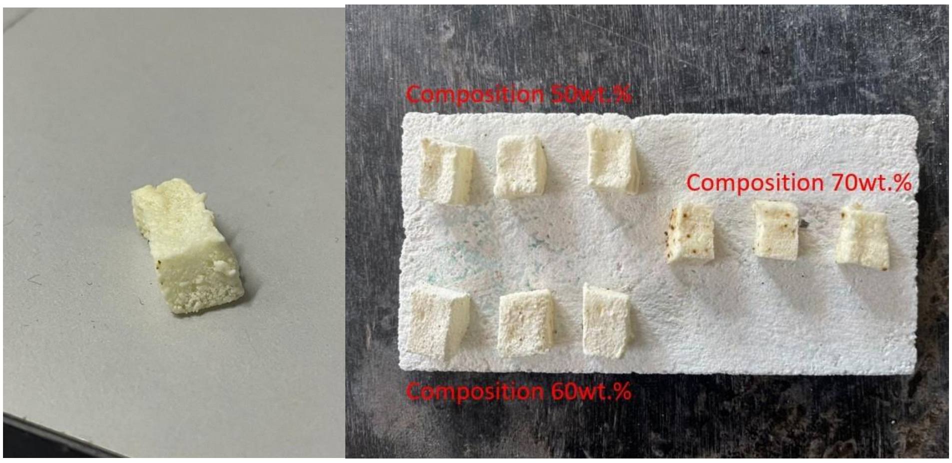
Hydroxyapatite binders, such as carboxymethyl cellulose (CMC) and polyethylene glycol (PEG), have a variety of mechanical and rheological qualities that make them suitable for a wide range of industrial and biomedical applications at compositions of 40 wt.%, 50 wt.%, 60 wt.%, and 70 wt.% (Fig. 6). Hydroxyapatite is mixed with two of these binders, carboxymethyl cellulose (CMC) and polyethylene glycol (PEG). These binders help improve HA's mechanical strength and processability, making it easier to shape and use in a range of applications, such as scaffolds, coatings, and composites. It became slurry after sintering in the Furnace in 1350 because using a CMC low mixture.

(a) **(b)**

Fig. 6 *(a) HA Foam 50wt.%,60wt.%and 70wt.% (b) Slurry HA Foam 50wt.%,60wt.%and 70wt.%*

3.2 Properties HA Foam binder Bentonite

Because of their varied mechanical and rheological characteristics, hydroxyapatite binders made of 50%, 60%, or 70% bentonite (Fig. 7) and distilled water are appropriate for a wide range of industrial and biomedical applications. Because of its exceptional biocompatibility and likeness to bone mineral, hydroxyapatite (HA), a bioceramic material, is widely used in the biomedical area for applications such as bone regeneration and dental repairs. Distilled water and binders like Bentonite are frequently used with HA to enhance its processability and mechanical qualities. Distilled water makes it easier to process and combine the binder uniformly, while bentonite clay improves the binding qualities. After sintering at 1300 C, changes in the weight percentages (wt%) of these components can have a substantial impact on the final HA binder's characteristics. That's why with the mixture of HA binder bentonite continued with the next three experiments to study more deeply about the material.

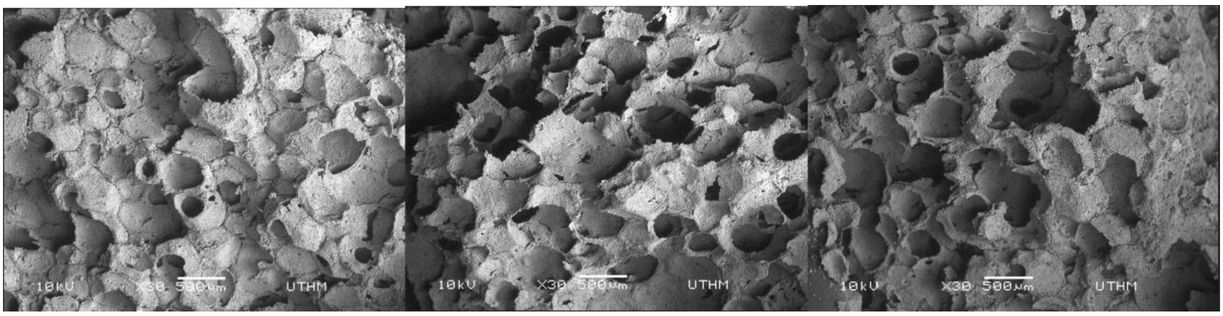


(a) (b)

Fig. 7 *(a) HA Foam binder Bentonite (b) HA Foam binder Bentonite 50 wt.%,60 wt.% and 70 wt.%*

3.3 Microstructural Analysis

The average pore widths for both closed and open pores in hydroxyapatite foam with compositions of 50 wt%, 60 wt%, and 70 wt%. The low concentration of the slurry prevents it from penetrating the foam completely, the 50wt. hydroxyapatite foam bonded with Bentonite has more closed pores. On the other hand, following sintering, the 60% composition, which has a more viscous slurry, develops wider open holes. Similar to this, the increased slurry viscosity in the 70% composition also results in bigger open holes. Comparing the compositions of 50 weight per cent and 70 weight per cent, the former exhibits larger closed pores overall.



(a) (b) (c)

Fig. 8 *(a) HA Foam 50wt.% Pores (b) HA Foam 60wt.% Pores (c) HA Foam 70wt.% Pores*

3.4 Density and Porosity Analysis

The table shows the porosity and density of hydroxyapatite foam samples with 50%, 60%, and 70% of hydroxyapatite compositions. Sample 2 has a higher density of 0.907 g/cm^3 and a porosity of 63.36%, while Sample 1 has a density of 0.772 g/cm^3 and a porosity of 59.38% for the 50wt.% composition. In the 60wt.% composition, sample 1 shows a porosity of 47.56% and a density of 1.134 g/cm^3, while sample 2 has a porosity of 51.78% and a higher density of 1.578 g/cm^3. For the 70wt.% composition, sample 1 has a porosity of 40.00% and a density of 1.220 g/cm^3, whereas sample 2 has the lowest density of 0.889 g/cm^3 and the maximum porosity of 81.90%. These variations demonstrate how changes in the hydroxyapatite composition and sample preparation affect the porosity and density of the foam, with the exception of the second sample's very high porosity of 70% weight. Higher density and reduced porosity are often the outcomes of higher hydroxyapatite content.

Table 1 *Density and Porosity for HA binder Bentonite*

Composition	Sample	Porosity Percentage (%)	Density (g/mm^3)
50wt.%	1	59.38	0.772
	2	63.36	0.907
60wt.%	1	47.56	1.134
	2	51.78	1.578
70wt.%	1	40.00	1.220
	2	81.90	0.889

3.5 Compression Testing

In conclusion, hydroxyapatite (HA) is used in HA foam composites at three different weight percentages (wt%): 50, 60, and 70%. These foams undergo compression testing to determine their mechanical strength and structural integrity under load. Most of the results indicate that when the weight percentage of hydroxyapatite increases, the foam's compressive strength increases as well. The 70 weight percent foam has the highest compressive strength of the two HA foams; the 60 and 50 weight percent foams have lesser compressive strengths. The reason for the improved strength of the 70% HA foam is its higher hydroxyapatite content, which provides more structural support and boosts the foam's ability to withstand greater compressive pressures.

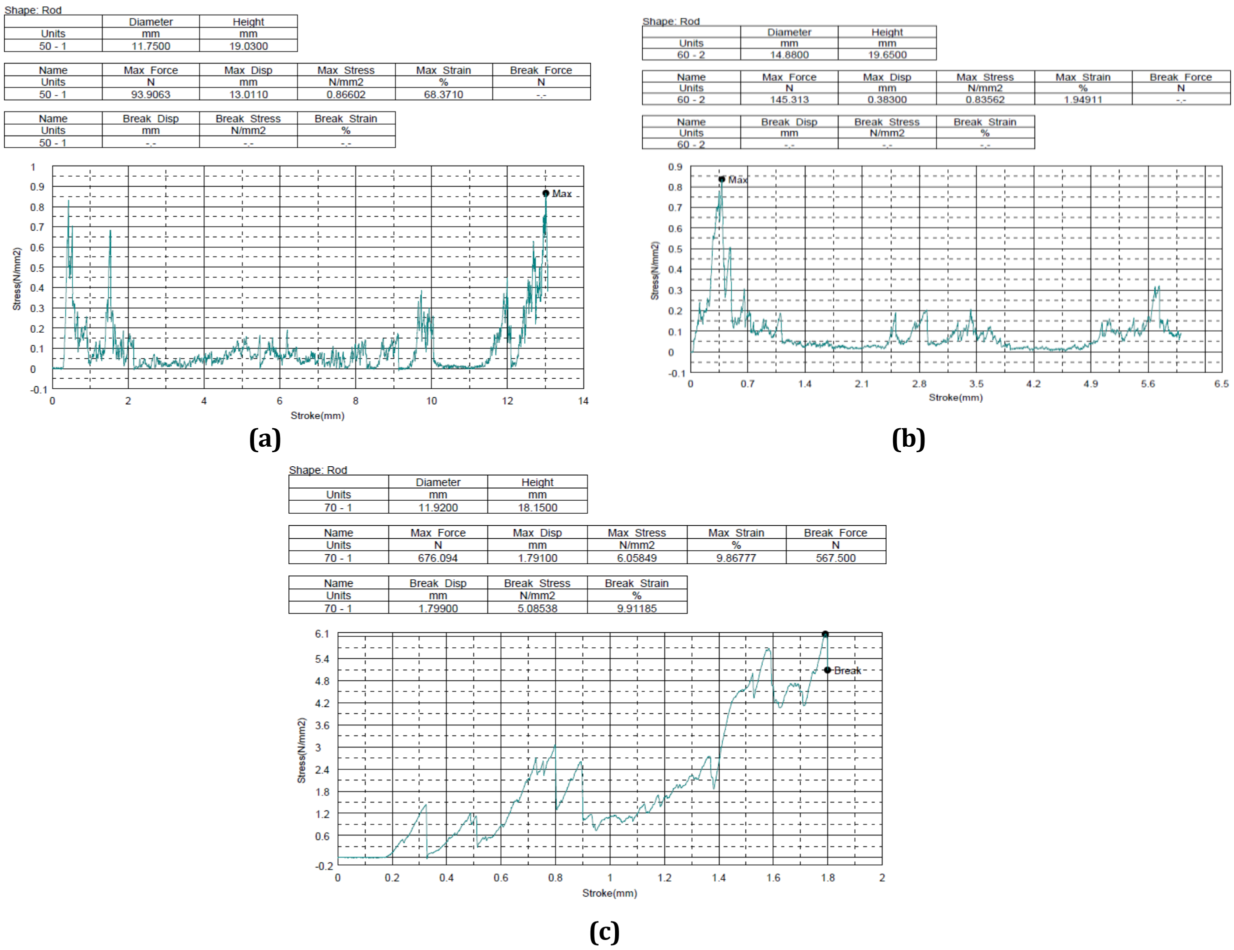


Fig. 9 *(a) Max Stress 50wt.% HA Foam, (b) Max Stress 60wt.% HA Foam (c) Max Stress 70wt.% HA Foam*

3.5 Summary

Compared to CMC (carboxymethyl cellulose) and PEG (polyethylene glycol), HA foam binder, or hydroxypropyl cellulose, shows better binding qualities with bentonite. This is because HA strengthens the overall stability and integrity of the foam structure by forming a more cohesive and stickier link with the bentonite particles. In contrast to CMC and PEG, HA interacts with the bentonite surface more successfully, forging a stronger network that withstands deformation and raises the foam's mechanical strength. Because of this, HA is the material of choice for applications where excellent bentonite and foam bonding is essential to longevity and performance.

4. Conclusion

When used as a bentonite binder in foam applications, hydroxypropyl cellulose (HA) outperforms both polyethylene glycol (PEG) and carboxymethyl cellulose (CMC). Strong bonds between HA and bentonite particles are formed with remarkable efficiency, greatly increasing the mechanical strength and stability of foam formations. In contrast to PEG and CMC, which frequently don't have the cohesive strength required for demanding applications, HA forms a cohesive network by successfully binding bentonite particles together. Because of its chemical makeup, HA sticks to bentonite surfaces more readily, increasing the overall stability of the foam. Furthermore, improved binding properties of HA lead to better foam performance under a variety of circumstances, such as moisture stability and long-term deformation resistance. Because of these qualities, HA is perfect for sectors that depend on sturdy foam products, like packaging, environmental cleanup, and construction.

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