Low-cost synthesis of hydroxyapatite on different bed materials: crystalline structure, morphology and process efficiency

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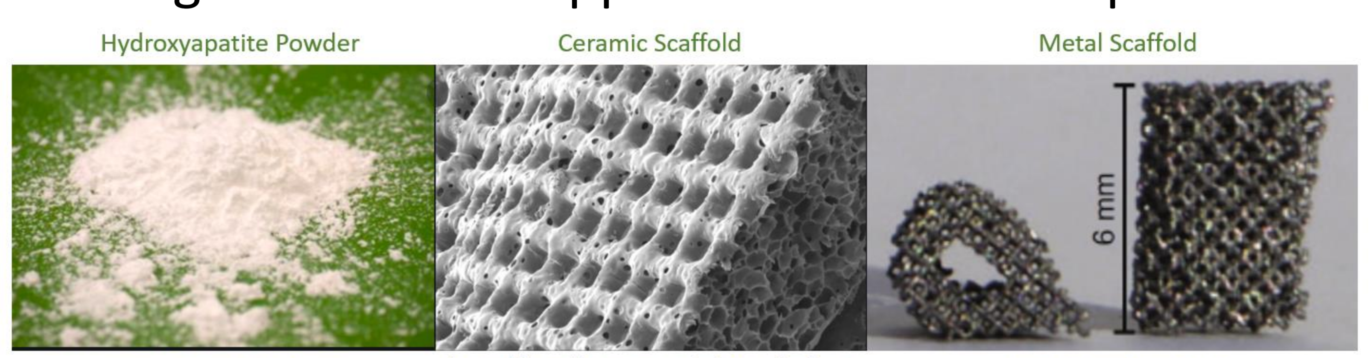
Abstract

- In this research, the effect of drying bed material (glass, aluminum and titanium) on the amount of residue and crystalline characteristics of the HA powder was studied. Hydroxyapatite (HA) powder was synthesized via sol-gel method and dried in oven at 150° C for an hour. Nano crystalline pure hydroxyapatite and biphasic hydroxyapatite could be synthesized from one HA sol-gel. The smallest residue percentage was 1.8%, for pure HA powder dried on aluminum bed. Bed materials did not affect crystal structure and size. Wetting angle, which is related to surface tension between liquid HA and solid drying bed, was found to be a key factor to control the amount of residue.

Introduction

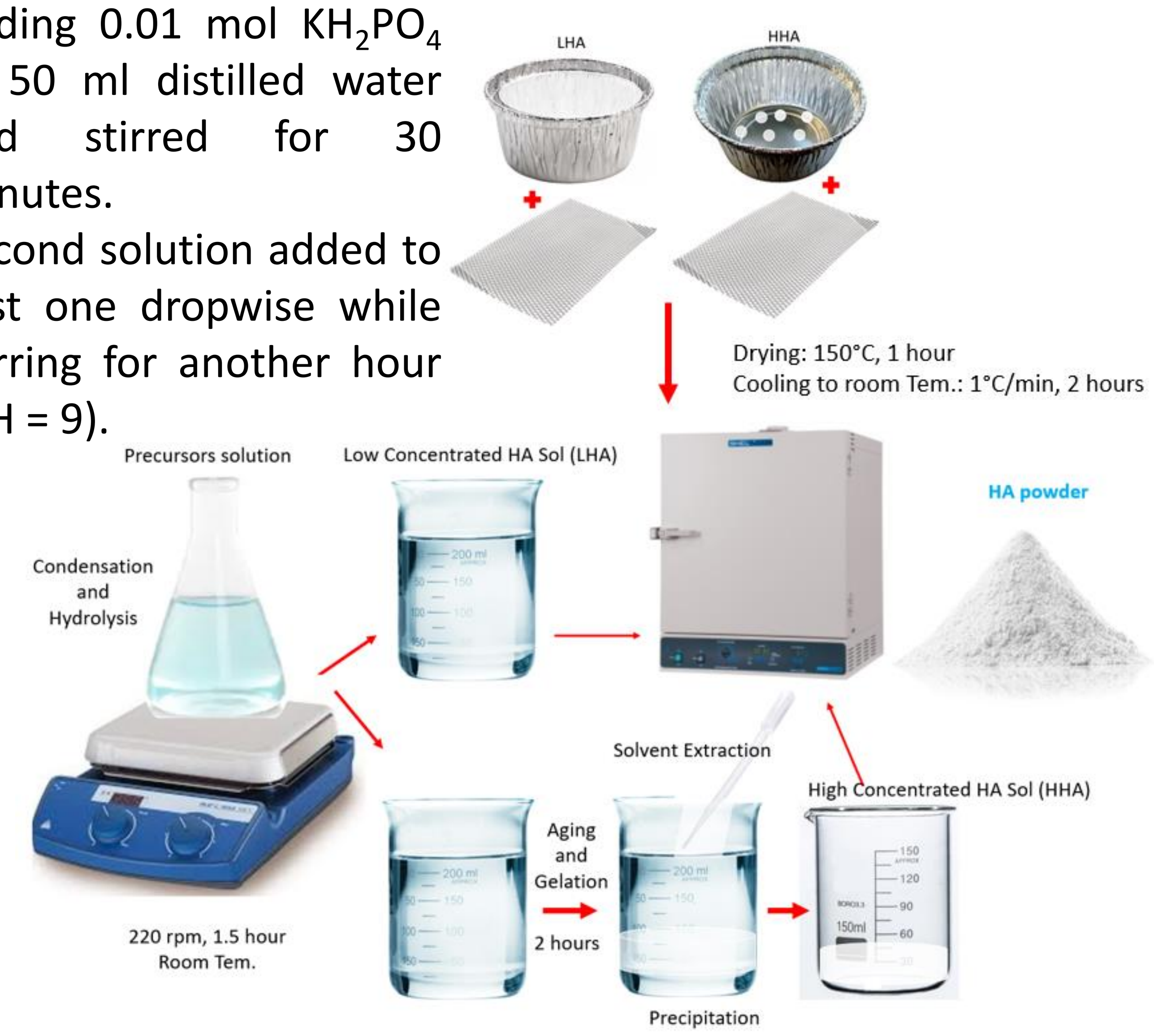
- Hydroxyapatite (HA, $Ca_{10}(PO_4)_6(OH)_2$) is one of the main calcium orthophosphate compounds, which has excellent biocompatibility.
- HA powder can be used in polymer/ceramic or metallic/ceramic scaffolds to promote bone regeneration for applications such as implants.



- During synthesis, there is always an amount of loss, remains or stains of HA on the drying bed, which increase production cost and decrease efficiency.
- High temperature and cooling systems in HA powder synthesis is an expensive procedure and consequently, raises the production and customers' costs.
- Objective of this work: produce low-cost HA with minimized residue and higher efficiency by sol-gel method on different bed materials, aluminum, glass and titanium.

Powder synthesis

1. Hydroxyapatite (HA) sols prepared: mixing two precursor solutions to maintain Ca/P ratio at 1.67.
2. $Ca(NO_3)_2.4H_2O$ (0.0167 mol) dissolved in 50 ml distilled water.
3. NH_4OH (2.5 ml) added dropwise to adjust pH around 12 while stirring for 30 minutes.
4. Second solution made by adding 0.01 mol KH_2PO_4 in 50 ml distilled water and stirred for 30 minutes.
5. Second solution added to first one dropwise while stirring for another hour (pH = 9).



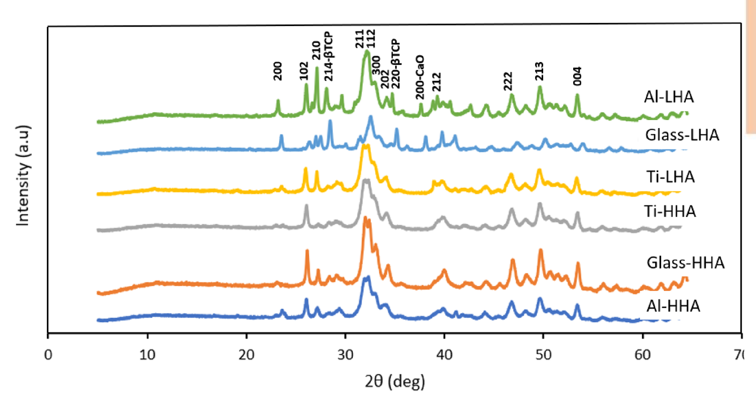
Characterization methods

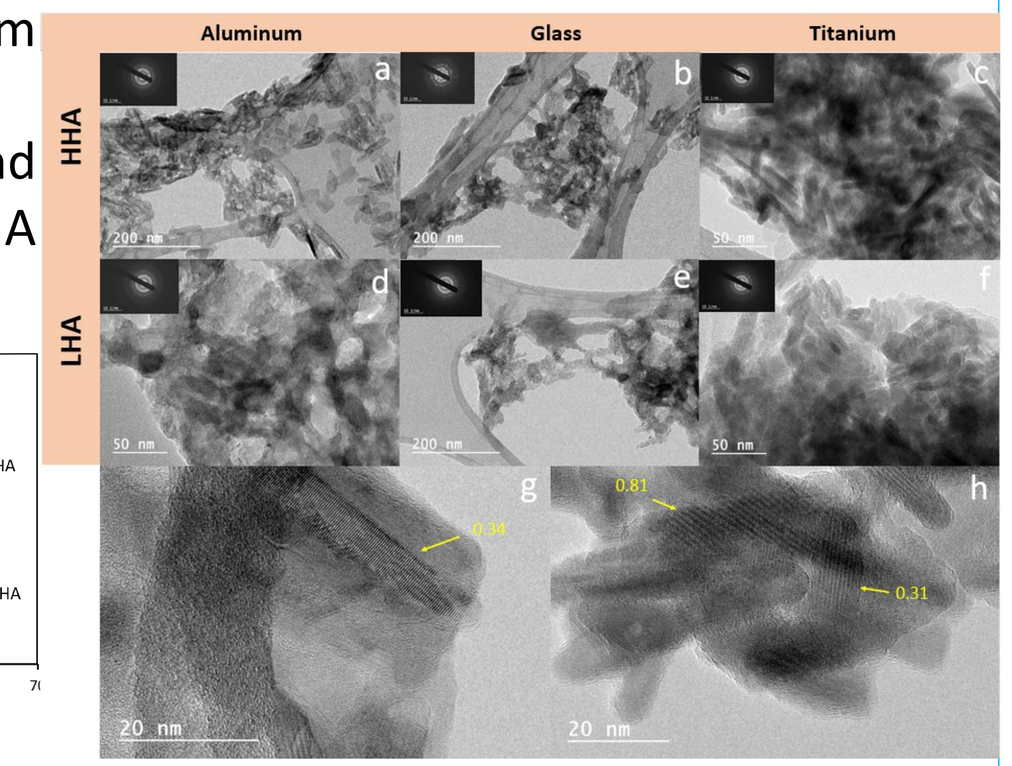
- XRD (X-ray diffraction pattern)
  - Crystal structure and composition
- TEM (transmission electron microscope)
  - Crystal shape and size
- FESEM (field emission electron microscope)
  - Morphology and porosity
- EDX (energy-dispersive X-ray spectroscopy)
  - Elemental distribution
- Contact angle measurement
  - Effect of surface tension on powder residue and method efficiency

Experimental Results

1- Structural and compositional analysis

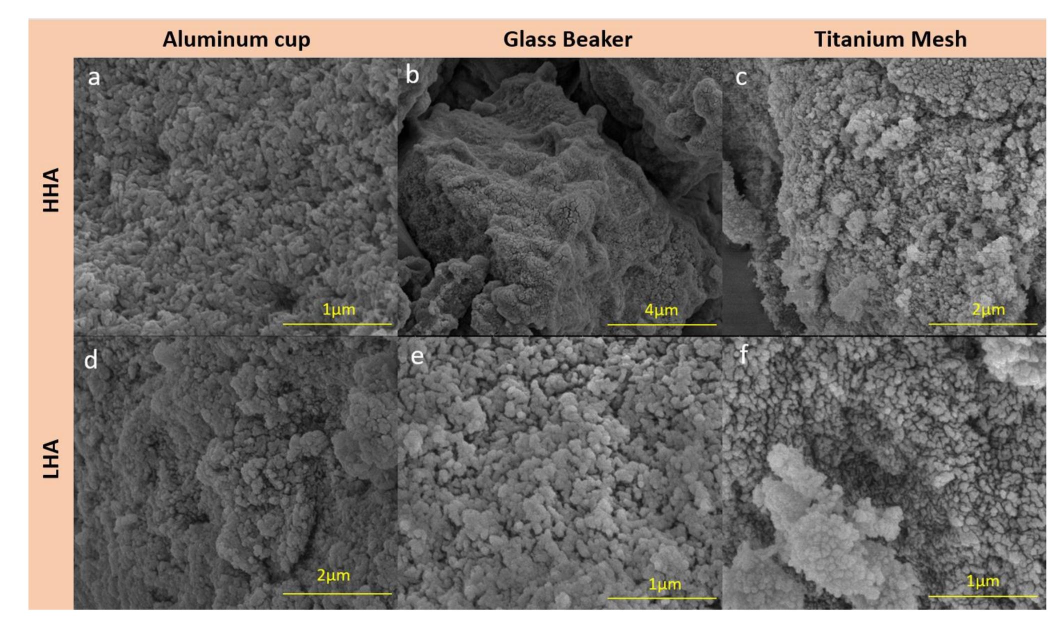
- XRD shows pure HA as major phase with β-TCP and CaO from LHA derived from glass beaker and aluminum cup.
- Nanorods pure HA as major phase with length 20-80 nm and diameter 5-10 nm were derived from HHA on all bed and LHA on titanium.
- The most brilliant planes for pure HA are d = 0.34 nm (002), d = 0.31 nm (102), and d = 0.81 nm (100).
- Crystal structure and size depend on initial sol and drying temperature.

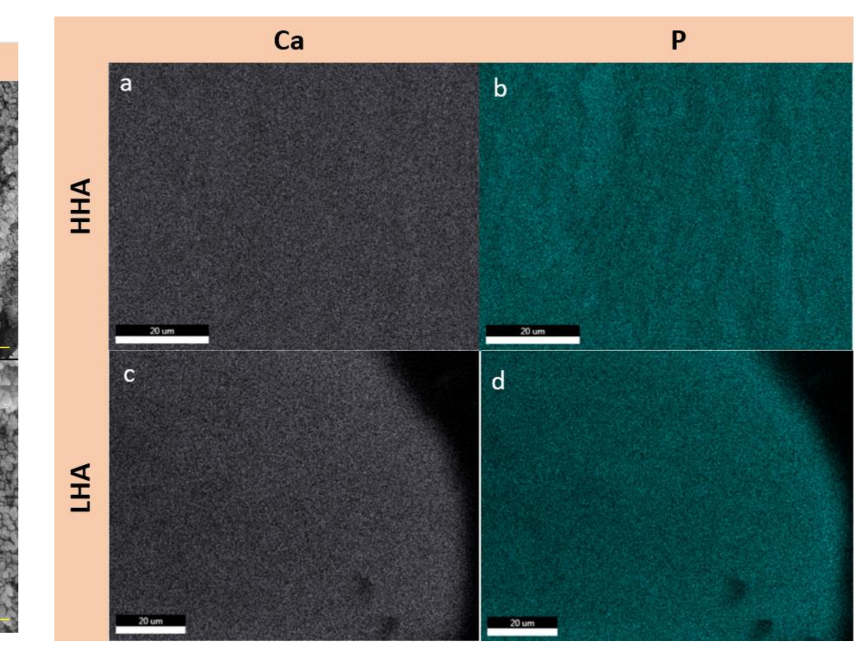




2- Morphology and elemental distribution

- Higher thermal conductivity leads to more nucleation sites on bed surface for sol, creating a powder with denser structure and less porosity (Aluminum).





- Powder with same structure, but with different porosity derived on all beds.
- Homogeneous elemental distribution for Ca and P in HA powder from HHA and LHA sol.

3- Contact angle and residue%

- Wetting contact angle is a key factor to determine residue.
- 98% efficiency obtained for HHA powder dried on aluminum cup.
- Titanium mesh has the least efficiency and highest residue due to surface morphology of holes and wires.

Bed substrate	Initial contact angle		Wetting contact angle		Mass residue %	
	HHA	LHA	HHA	LHA	HHA	LHA
Aluminum cup	77°	0°	67°	0°	1.8	34
Glass beaker	65°	49°	0°	0°	45	49
Titanium mesh	93°	118°	0°	0°	76	73

Conclusion

- Nanorods pure and biphasic hydroxyapatite with low porosity were derived from precipitated and calcium deficient HA sol (HHA and LHA) at low temperature in about 7 hours from all beds.
- Best efficiency was 98%, obtained on aluminum bed for high concentration sol.
- Titanium mesh substrate acted as a filter, which can synthesize pure HA from both precipitated and calcium deficient HA sol at 150°C.
- Glass beaker, which is the most popular substrate, led to about 50% efficiency for both sol.

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