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A water-soluble core material for manufacturing hollow composite sections



Z. Xiao, L.T. Harper*, A.R. Kennedy, N.A. Warrior

Composites Research Group, Faculty of Engineering, University of Nottingham, NG7 2RD, UK

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ABSTRACT

This paper presents the development of a low-cost water-soluble core material, which is suitable for producing hollow composite structures via high pressure moulding processes, such as compression moulding and resin transfer moulding. The bulk material of the core is sodium chloride (NaCl), which is held together by a water-soluble trehalose binder. The composition of the core has been optimised to provide acceptable dissolution rates and mechanical properties for high volume structural composite applications.

The compressive strength of the NaCl core was 57 MPa at ambient temperature, which reduced to 20 MPa when tested at 120 °C. The compressive strength at elevated temperature was approximately 4 times higher than for a water-soluble commercial benchmark and 33 times higher than a conventional structural closed-cell foam. The specific dissolution rate of the NaCl core was between 0.14 and 1.23 kg/(min·m²), depending on processing parameters and the coefficient of thermal expansion was approximately 43×10^{-6} /K. A practical example has been presented to demonstrate how the removable core can be used to produce a representative hollow section of an integrally stiffened panel.

1. Introduction

Core materials are used to separate structural composite skins in order to increase the second moment of area and greatly improve the bending stiffness. Foam materials are commonly used to form sandwich panels, but current high volume composite manufacturing processes, such as compression moulding or high pressure resin transfer moulding (HP-RTM), can generate pressures of 30–150 bar inside the mould tool [1,2]. Closed-cell structural foams therefore typically require very high densities (for example 200 kg/m³ for a compressive strength of 6 MPa [3]) to prevent crushing in many applications. These materials tend to stay in the final moulding and therefore add parasitic mass.

Removable core materials are available and are commonly used for manufacturing hollow metallic structures, for example sand cores for casting automotive cylinder blocks [4,5]. Sand cores are held together by a binder material such as sodium silicate, which hardens when exposed to carbon dioxide [6]. Mechanical removal methods, such as shaking and knocking, are required to extract the sand, which can be difficult if access is limited or the component is fragile, as the bonded sand particles do not readily flow [6]. Plastics such as polyoxymethylene (POM) and low melting temperature metals such as zinc based alloys (ZnAl₄Cu₁) are also used to make cores, and can be burnt off or melted out at elevated temperatures [4]. The removal temperatures for extractable polymers (~180 °C) and metals (380 °C) are typically incompatible with the polymer matrix, or they are insufficiently stiff and can crush under the moulding pressure. Caustic solutions containing sodium hydroxide may be used for removing ceramic-based cores [7,8], but this is harmful to the environment and can affect the mechanical performance of the finished product [9]. Current removable cores tend to be incompatible with high pressure moulding processes, as the resin can infiltrate into the core during the infusion process, impeding the core removal. Clearly there are opportunities for reducing the mass of structural composite components, but the challenge is finding a removable core material that can withstand high pressures during the moulding process, is impermeable to the low-viscosity resin, can be quickly and easily removed and can be manufactured at low cost.

This paper investigates the potential for using water soluble sodium chloride (NaCl) cores for producing hollow carbon fibre composites, similar to the ones that are used to produce high pressure die castings [10]. The cores consist of NaCl and a water-soluble and environmentally friendly binder, trehalose sugar, which can be hot pressed into shape. They can be simply washed out after the composite moulding cycle using water and can potentially be recovered. A study is presented to define a processing window for the NaCl core to achieve suitable mechanical properties for use in high pressure moulding processes (100–150 bar), whilst ensuring the core can still be removed from the composite, post moulding. The influences of binder content, processing pressure, temperature and time are all investigated.

* Corresponding author.

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E-mail address: lee.harper@nottingham.ac.uk (L.T. Harper).

2. Background

The following criteria has been identified to ensure the core material is suitable for high pressure composite moulding processes:

- Sufficient compressive strength (typically 15 MPa) at elevated mould temperatures (typically 120 °C).
- Impermeable to low viscosity resins.
- Fully removable and within the Takt time of the moulding process (typically 5–30 min).
- Easily removable without inducing damage to the composite structure.
- Machineable or castable to produce complex geometries.
- Low cost.
- Recyclable and environmentally friendly.

Inorganic salts are an appropriate material choice, as commercial salt cores are reported to have flexural strengths of over 18 MPa [10] and have been used for high pressure die casting (HPDC) at temperatures and pressures exceeding 690 °C [4] and 60 MPa [11] respectively. Different compositions have been investigated in the literature, including KCl based cores for high bending strength at elevated temperatures [4,12], NaCl cores containing mica powder for cost reduction [7], NaHCO₃ based cores for accelerating core removal [13], and four binary systems (NaCl-Na₂CO₃, KCl-K₂CO₃, KCl-NaCl and K₂CO₃-Na₂CO₃) for increasing mechanical properties compared with NaCl cores [11]. The material is readily available and inexpensive and can be dissolved in water to enable the core to be washed out from the composite structure. There is also the potential to recover some salt from the waste solution.

There are typically three binding mechanisms for salt cores. These include 1) mechanical pressing of salt grains followed by sintering, 2) melting followed by consolidation and 3) adhesives (see Table 1). For sintering, salt particles are typically pressed at over 100 MPa and then thermally treated at 650 °C to increase the bending strength from 5 MPa to 9 MPa [4]. Whilst sintered cores have good mechanical properties and low porosity (reported as 2%-6% [4]), they are limited to producing simple geometries because a non-hydrostatic pressure is generated in the tool. The magnitude of the processing pressure is also an issue for larger scale components, due to the high capital costs for tooling and a hydraulic press. For the same reasons, casting liquid salt cores is not appropriate for large composite components. In addition, the energy consumption for producing cast liquid salt cores is high due to the high heat capacity and melting point of salt (800 °C) and the shrinkage of the core can be significant (> 15 vol%) following solidification [4,11]. The dissolution rate for dense salt cores is also very slow, requiring high pressure water jets (600 bar) to remove the core [14].

Using a binder to stabilise the salt core can effectively reduce the required consolidation pressure to less than 1 MPa [7], as a high level of mechanical interlocking of the salt grains is not required to achieve acceptable properties. The mixture can also be pressed warm or cold, depending on the binder used [4,7,12,13]. Binders used for salt cores are often sodium silicate (water glass), synthetic resins, and polyethylene glycol [4,7,12,13]. Compared to sintered cores, more complex shapes can be produced but the porosity level is much higher (up to

30% [4]), and therefore mechanical properties are lower. Depending on the binder type, the addition of such a parasitic material can have a negative effect on the dissolution rate [4].

3. Salt core development

For the current work, extra fine NaCl powder with an average particle size of 0.59 mm was used to manufacture the cores, referred to here as NT Cores. Trehalose sugar was used as the binder material, which had a powder particle size of < 0.30 mm. Trehalose is a nonreducing sugar formed from two glucose units joined by a 1-1 alpha bond. It is therefore water-soluble, compatible with NaCl and does not appear to affect the fibre reinforced composite. The powders were mechanically mixed to the required ratio and then poured into a preheated cylindrical mould with an inner diameter of 150 mm. The mould surface was coated with Frekote 700-NC mould release to aid removal of the core. The salt/sugar mixture was hot compacted and held at temperature, before the tool was cooled in the press at a rate of 5 $^{\circ}C/$ min. The core was demoulded when the tool temperature had reduced to 30 °C. Test coupons were machined from the round disc, typically 13 mm thick, using a 3 mm wide diamond tipped circular saw (see cutting plan in Fig. 1). Before machining, the dimensions and mass of each core were recorded to calculate the density.

3.1. Design of experiment

A Taguchi study was conducted to establish the relative significance of a range of processing variables on the strength of the NT Core at ambient temperature, and the dissolution behaviour in water. The independent variables and their associated levels are summarised in Table 2. Processing pressure directly influences the cost of the tooling and capital expenditure and therefore should be minimised to produce cost effective cores for high volume applications. The effect of binder content, processing temperature and processing time were all investigated as possible ways to compensate for lower processing pressures.

With a broad "processing window" defined, a more detailed second study was conducted to determine the effect of elevated in-service mould temperatures, with tests conducted at 80 °C and 120 °C. Two widely-used core materials were also tested as benchmarks: a structural closed cell foam (A Core) and a water-soluble material containing ceramic microspheres (B Core). The B Core material was moulded in the same circular tool as the NT Cores at a pressure of 17 bar at room temperature. The demoulded cores were subsequently dried in an oven at 125 °C for 1.5 h.

3.2. Experimental methods

3.2.1.Mechanical testing

Both 3-point bend and compression tests were conducted on an INSTRON 5985 universal testing machine. For 3-point bending tests, five $13 \times 13 \times 100$ mm specimens were prepared for each material and a constant span of 60 mm was used between Ø10 mm support rollers for all specimens. Tests were performed at a speed of 1 mm/min. The applied force was recorded by a 25 kN load cell and the central

Table 1

Salt cores made by different processes and their mechanical property.

| Manufacturing process | Core material | Flexural strength (MPa) | Comments |
|-----------------------|--------------------------------------|-------------------------|---|
| Sintering | KCl NaCl | > 9.0 | Pressed at 104 MPa; Additives: ZrSiO ₄ , Al ₂ O ₃ , SiO ₂ , Fe ₂ O ₃ ; Heat treatment at 650 $^\circ C$ [4] |
| Binder | NaCl | 2.1 | Binder: water glass; Pressure: 7.5-8 bar [4] |
| | NaCl | 6.6 | Binder: polyethylene glycol and 10 wt% plasticizer; Pressure: 4-5 bar [7] |
| Liquid casting | NaCl | > 18.0 | [14] |
| | NaCl Na ₂ CO ₃ | > 30.0 | NaCl-70 mol% Na ₂ CO ₃ [11] |



Fig. 1. Cutting plan of test specimens from a circular disc, 150 mm diameter, 13 mm thick.

Table 2

Parameters and associated levels in the Taguchi study.

| Parameters | Levels | | |
|-----------------------------|--------|-----|-----|
| Binder content (wt%) | 3 | 5 | 8 |
| Processing pressure (bar) | 55 | 85 | 170 |
| Processing temperature (°C) | 120 | 150 | 190 |
| Processing time (min) | 10 | 30 | 60 |
| 0 | | | |

deflection of the specimen was recorded by an LVDT positioned on the underside of the specimen. Three compression test specimens were prepared for each material configuration, which were cut to $30 \times 30 \times 13$ mm and compressed between Ø50 mm platens at a speed of 1 mm/min. A 100 kN load cell was used to record the applied compressive force.

3.2.2. Microscopy

The cross-sections of the core materials were observed by optical micrographs at $5 \times$ magnification. The specimens were cut from the ends of the flexural test specimens after the bending test was finished, and machined into $13 \times 13 \times 13$ mm cubes. The faces of interest were polished unmounted, using silicon carbide paper (P240, P400, P800, P1200, P2500, and P4000) but without using any fluids.

3.2.3. Thermoanalytical testing

Coefficient of thermal expansion (CTE) measurements were conducted on a Q400 TMA from TA Instruments. CTE measurements were performed on the microscopy specimens, across the temperature range from 20 $^{\circ}$ C to 120 $^{\circ}$ C.

Differential scanning calorimetry (DSC) was conducted for the trehalose binder on a Q100, supplied by TA Instruments. For the as-received trehalose powders, a full profile scan was performed at a rate of 5 °C/min from 20 °C to 230 °C. To represent the core manufacturing conditions, the trehalose was heated at 5 °C/min from 20 °C to 150 °C and held at 150 °C for 1 h, before being cooled to 20 °C. Subsequently, the same sample was reheated up to 150 °C at the same ramp rate to identify whether the trehalose had experienced a phase change after the core had been made.

3.2.4. Dissolution rate measurements

An ultrasonic water bath (Sonic 3000 from James Products Ltd) and

a conductivity meter (HANNA HI8733) were used to measure the dissolution rate of the core. A calibration line was created by measuring the conductivity of known quantities of NaCl in one litre of distilled water at 25 °C. NT Core specimens of $13 \times 13 \times 20$ mm were used for the dissolution rate measurements. Each specimen was weighed and then positioned in the middle of the water bath inside a wire basket. The conductivity of the water was recorded from the point at which the specimen was placed in the water until it reached a constant value (indicating that the sample had fully dissolved). The change in conductivity was converted into the change of NaCl concentration using the reference line. The dissolution rate of the core material, d_n (kg/ min), was calculated using the gradient of the concentration change curve. The specific dissolution rate, d_s (kg/(min·m²)), was calculated by:

$$d_s = \frac{d_n}{A} = \frac{\Delta C \cdot V}{A \cdot \Delta t} (1 + \varepsilon) \tag{1}$$

where Δt is the time period, ΔC is the concentration change during time period Δt , *V* is the volume of water, ε is the binder content of each sample, and *A* is the total surface area of each specimen.

3.2.5. Machining trials

Machining trials were performed to observe the effects of cutter type on the quality of the machined surface. A 4-flute ball nose cutter and a 4-flute flat end mill, both with a diameter of 10 mm, were selected to make slots in core specimens. A spindle speed of 1400 rpm was used with a feed speed of 200 mm/min to produce a 1 mm deep cut. Five consecutive cuts were completed to produce a 5 mm deep slot. Surface roughness profiles were measured at different locations along the slots using a Talysurf CLI 1000 mounted with an inductive gauge. Fig. 2 indicates the locations of the measurements along the two different shaped slots.

3.2.6. Core removal trials

Water jet removal trials were conducted for a B Core sample and an NT Core sample (same composition as used for the machining trials). Both core materials were machined into trapezoidal columns, shown in Fig. 3(a). To ensure a direct comparison, all cores were coated with 3 layers of Aquaseal 3818 coating (made by Advanced Ceramics Manufacturing). Each layer of Aquaseal was sprayed on the core, which was then allowed to dry in an oven at 40 °C for 30 min before the next layer was applied. Cores were then wrapped with two plies of MTM 49 – 3/CF1218 twill weave T800 carbon fibre prepreg from Cytec, keeping one end open to facilitate the removal of the core (Fig. 3(b)). This whole arrangement was placed on a metallic plate, vacuum bagged and put into an autoclave at 100 °C and 6 bar for 4 h to cure.

The cured samples were removed from the autoclave and clamped into a fixture to maintain a constant 10 mm distance between the open end of the sample and the water jet (Fig. 3(c)). The nozzle had an elliptical orifice (the lengths of the semi-major and semi-minor axes were 1 mm and 0.75 mm respectively) and a 25° spray angle. A water temperature of 40 °C and a pressure of 8 bar were used for all tests. Each test was stopped after 10 s intervals to record the amount of core removed. The residual water in the hole was poured into a measuring cylinder to record the volume, to indicate the volume change of the core.

4. Water-soluble core characterisation

4.1. Core strength at ambient

Both the compressive and flexural strengths of the NT Core follow similar trends as the four main parameters are increased, as shown in Table 3. The most significant variable affecting both strengths was the applied pressure during the compaction process. The average compressive strength was 17 MPa for all samples moulded at a pressure of 1 - Top surface;



3 - Cutting surface/ Ball-nose cutter;

6 - Inner cutting edge/Flat cutter;

Fig. 2. Schematics of two types of cutting slots, scanning direction and range of 50 mm (a); 6 locations where the inductive gauge places (b) on a round sample disc with 150 mm diameter.

55 bar. An increase in moulding pressure from 55 bar to 85 bar increased the compressive strength on average, by 19%. A further increase in moulding pressure to 170 bar increased the compressive strength by 144% over the average value observed at 55 bar. Increasing the processing pressure reduces the free space between the powder particles, strengthening the mechanical interlocking of the angular salt grains.

4 – Cutting edge/Flat cutter; 5 – Cutting surface/Flat cutter;

2 - Cutting edge/Ball-nose cutter;

Adding binder significantly improves the mechanical performance of these core materials, as the binder provides some adhesion. Dispersion of the binder improves as the binder content increases, increasing the number of salt to binder contact points. The average compressive strength increased by 16% as the binder content was increased from 3 wt% to 5 wt%, and by 28% as the binder content increased to 8 wt%. A similar increase in flexural strength was observed with increasing binder content. Fig. 4 shows compressive and flexural stress/strain curves for cores with different binder levels. The shape of the compression curves are similar for increasing binder content, but the shape of the flexural curves changes as the core becomes more brittle (lower tensile properties compared to compressive properties) with increasing binder levels (when processed at 190 °C). This demonstrates that the binder content influences the failure mode. Mechanical inter-locking of the salt particles is responsible for binding the core together at low trehalose levels. The salt particles can slip relative to each other when a load is applied, exhibiting plastic-like behaviour. The strain to failure decreases as the trehalose content increases and a linear stress/strain curve is observed for an 8 wt% loading, as the failure of the core is due to binder fracture.

The failure mode also changes as the core is processed at different temperatures, as the binder goes through different phases. Fig. 4(b) shows that cores manufactured at over 150 °C exhibit brittle behaviour in bending, compared to a more ductile failure mode when processed at lower temperatures. According to Table 3, the mechanical properties of the cores were improved with increasing processing temperature and time, since different phases of the binder were formed and the level of phase transformation was higher, as evidenced by the changes in colour of the samples. Phase characterisation of the trehalose is discussed in detail in the following section.

4.2. Phase characterisation of trehalose

Fig. 5 shows a DSC curve for the trehalose binder, indicating heat flow in the temperature range of 20 °C–230 °C, which is consistent with data in the literature [15,16]. Two endothermic peaks were observed in the thermal profile at 100 °C and 125 °C, and a twin-peak was found at 185 °C–210 °C. All peaks were labelled according to Sussich et al. [16]. Trehalose has three crystalline forms during the core moulding cycle, as





Closed end

Table 3

All experimental results in Taguchi study showing the mean effects of four independent variables (pressure, temperature, time and binder content) on four dependent variables (compressive strength, flexural strength, density and SDR).

| Mean | Processing pressure (bar) | | Binder content (wt%) | | Processing temperature (°C) | | | Processing time (min) | | | | |
|--------------------------------|---------------------------|-------|----------------------|-------|-----------------------------|-------|-------|-----------------------|-------|-------|-------|-------|
| | 55 | 85 | 170 | 3 | 5 | 8 | 120 | 150 | 190 | 10 | 30 | 60 |
| Density (g/cm ³) | 1.48 | 1.54 | 1.77 | 1.59 | 1.60 | 1.61 | 1.55 | 1.61 | 1.64 | 1.56 | 1.60 | 1.64 |
| Change ratio | - | 4% | 20% | - | 0.6% | 1.2% | - | 4% | 6% | - | 3% | 5% |
| Comp strength (MPa) | 16.92 | 20.16 | 41.22 | 22.78 | 26.42 | 29.10 | 23.37 | 26.19 | 28.74 | 23.99 | 25.72 | 28.60 |
| Change ratio | - | 19% | 144% | - | 16% | 28% | - | 17% | 28% | - | 7% | 19% |
| Flexural strength (MPa) | 3.92 | 4.96 | 7.92 | 4.74 | 5.75 | 6.30 | 5.20 | 5.81 | 6.34 | 4.62 | 5.58 | 6.48 |
| Change ratio | - | 27% | 102% | - | 21% | 33% | - | 10% | 22% | - | 18% | 37% |
| SDR (kg/(min·m ²)) | 0.71 | 0.63 | 0.42 | 0.64 | 0.62 | 0.50 | 0.84 | 0.58 | 0.34 | 0.80 | 0.61 | 0.35 |
| Change ratio | - | -11% | -41% | - | -3% | -22% | - | -31% | -60% | - | -24% | - 56% |

the ramp rate is over 5 °C/min: T_h (as-received, dihydrate), T_γ (metastable anhydrate) and T_β (stable anhydrate). This is in agreement with Sussich et al. [16], who reported the formation of a stable anhydrate (T_β) during the amorphous state between 150 °C and 180 °C when a fast heating rate was used (> 10 °C/min). This phase transformation explains the observed change in bending behaviour of the cores (shown in Fig. 4(b)), as a more brittle failure occurs as the processing temperature

increases from 120 °C to 150 °C. Fig. 6 demonstrates that there are no further phase changes when the core is reheated to 150 °C, which implies that the binder is stable during the composite moulding process. This is because the trehalose becomes amorphous or a stable anhydrate at 150 °C (see Fig. 5) and any water is fully removed during the 1 h processing time. According to Furuki et al. [17], these two phases are both stable in a dry environment. Based on the mechanical test results,

Fig. 4. Compressive (a) and flexural (b) stress/strain curves for NT Cores with different binder levels and processing temperatures (manufactured at 170 bar for 10 min).



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Fig. 5. DSC profile of trehalose from 20 $^\circ C$ to 230 $^\circ C$ (heating rate 5 $^\circ C/min).$



Fig. 6. DSC profiles of trehalose from two heating process in succession within 20 $^\circ\text{C}{-}150$ $^\circ\text{C}{.}$

the stiffness of the cores is significantly improved when the trehalose is processed at > 150 °C, indicating that the amorphous and stable anhydrate states have a positive effect on the mechanical properties.

Taylor et al. [15] and Sussich et al. [16] observed a state change for the dihydrate trehalose during heating. The trehalose is a solid when the applied temperature is below 120 °C, but it starts to become tacky as the temperature exceeds 140 °C and dehydration occurs. The trehalose turns to a fully liquid state at approximately 200 °C. Fig. 7 shows the distribution and morphology of the trehalose binder relative to the salt for an 8 wt% loading, processed at two different temperature levels. The binder remains in a granular state at low processing temperatures and porosity is clearly visible. Conversely, at higher processing temperatures the softened binder migrates into the voids between the salt particles, which results in a reduction in porosity. Furthermore, this further explains why the mechanical performance improves when processing temperature increases.

4.3. Core density

The density of the NT Core follows a similar trend to the mechanical properties, as the four independent variables are increased (Table 3). The density of the core is also dominated by the applied pressure during processing, which increases the mechanical inter-locking and reduces the free-space between the salt particles. The density of the core increases by 20% as the pressure is increased from 55 bar to 170 bar. Reducing the void content reduces the likelihood of undesirable resin penetration into the core during the composite moulding process, reducing the amount of residual material left within the composite part. Resin wicking into the core also increases the risk of dry spots forming in the composite laminate if a pre-impregnated fibre system is used, as the reinforcing fibres become starved of resin. Density calculations indicate that the average residual porosity of NT Cores pressed at temperatures higher than 150 °C and pressures below 170 bar is approximately 10%, which is likely to be interconnected at this level. The target porosity level for avoiding resin infiltration into the core is 7% according to [18]. It may therefore be difficult to avoid resin



Fig. 7. Optical microscope images of cross sections of the NT Cores having 8 wt% binder content and processed under 170 bar at (a) 120 °C for 10 min (porosity:16%) and (b) 190 °C for 10 min (porosity: 10%).

penetration without a large increase in core processing pressure or binder content, and consequently it may be necessary to coat the soluble core with an impervious layer prior to use. Increasing the binder content from 3 wt% to 8 wt% has a limited effect on the density of the core, which increases by only 1.2%. This is because of the relative densities of these two materials (2.16 g/cm^3 compared to 1.58 g/cm³).

4.4. CTE measurement

Thermal expansion of the NT Cores is linear within the range of 20 °C–120 °C, with CTE values reported to be $42-44 \times 10^{-6}$ /K. According to [19], the CTE of pure NaCl increases from 39.80×10^{-6} to 42.90×10^{-6} /K over the same temperature range, which indicates that the CTE of the cores is dominated by the NaCl rather than the trehalose binder. Therefore, the four independent variables (pressure, temperature, time and binder content) do not significantly affect the CTE of the core. The CTE range for the NT Core is close to that of commercially available sandwich panel cores [20]. High levels of thermal expansion are beneficial for generating in-mould pressures to improve the consolidation of closed sections.

4.5. Specific dissolution rate (SDR) measurement

The degree of dissolution as a function of time has been plotted for each NT Core sample, based on the conductivity change, as shown in Fig. 8. It can be seen that the dissolution rate is approximately linear between 1 and 5 min, which equates to approximately 50% dissolution for most samples. The gradient of the line within this time range can be used to calculate the specific dissolution rate (SDR) using Eq. (1).

From both Fig. 8 and Table 3, it can be seen that the SDR of the NT Core decreases with increasing levels of all four of the main parameters listed in Table 2. All SDR values are in the range of 0.14-1.23 kg/ (min·m²). The SDR of the core which attained a flexural strength of 10.0 MPa (170 bar and at 150 °C for 30 min, 8 wt% binder) is 0.25 kg/ (min·m²), which is ~5 times faster than the SDRs of the NaCl/mica/ polyethylene glycol core in [7], which had a flexural strength of just 4.1 MPa. It is also ~140 times faster than the NaCl/mica/polyethylene glycol core with 10 wt% plasticiser, which exhibited a flexural strength of 6.6 MPa in Ref. [7]. Increasing the processing pressure and binder content both increase the density of the NT Core, causing a reduction in the interconnected porosity and therefore reducing the SDR. Processing temperature is the most significant variable in terms of changes to SDR, closely followed by processing time. The SDR of the core decreased by

60% when the temperature increased from 120 °C to 190 °C and 56% when the processing time increased from 10 min to 60 min. Increasing either processing temperature or time can lead to an increase in the density of the core as discussed above, which reduces the dissolution rate in water.

5. Process optimisation

5.1. Elevated temperature testing

The main observations from the Taguchi study have been used to design a full-factorial study to understand the influence of in-service temperatures on the flexural and compressive strengths of the NT Cores. A processing pressure of 170 bar and binder content of 8 wt% were selected, based on the discussion above. Figs. 9 and 10 show the compressive and flexural strengths of the NT Cores made using various processing temperatures and times, tested at 20 °C, 80 °C and 120 °C. The ultimate strengths were highest when the cores were tested at ambient temperature; 57.3 MPa for the compressive strength and 10.2 MPa for the flexural strength. This flexural strength value is ~ 2.5 times higher than the value presented by Jelinek et al. [4] for a NaClbased core pressed at 104 MPa, which is ~6 times higher than the pressure used in this work (17 MPa). Mica powder is commonly used as a reinforcement for NaCl [21] and was used in Ref. [7] to increase the strength of salt cores. However, the flexural strength was limited to just 6.7 MPa, 35% lower than the current NT Core system.

The general trends for the ambient temperature data in Figs. 9 and 10 are consistent with the data presented in the Taguchi study in Table 3. Both the flexural and compressive strengths increase with increasing processing temperature. However, there is a dramatic reduction in both the flexural and compressive strengths as the test temperature increases to 120 °C. (The reduction between 80 °C and 120 °C is less significant). The peak compressive strength is reduced from 57.3 MPa to 19.7 MPa and the peak flexural strength is reduced from 10.2 MPa to 3.7 MPa. Whilst there is a significant drop in mechanical performance at this temperature, the compressive properties of the core (15–20 MPa) are still sufficient for the application in HP-RTM and compression moulding.

The ultimate strengths are shown to increase between processing temperatures of 120 $^{\circ}$ C and 150 $^{\circ}$ C, even when tested at elevated temperatures. However, there is a reduction in properties as the processing temperature increases further to 190 $^{\circ}$ C and the processing time increases to 30 min. The flexural strength of the sample manufactured



Fig. 8. Degree of dissolution for NT Core samples in the Taguchi study (legend shows: processing pressure-binder content-processing temperature-processing time).

using a processing temperature of 190 °C for 30 min, and tested at 120 °C, is lower than the same sample processed at just 120 °C. This can be attributed to decomposition of the binder at elevated temperatures, particularly when the test temperature is close to the melting temperature of trehalose and longer processing times are used. At 190 °C the trehalose sugar starts to 'caramelise', i.e. the fructose and glucose in the sugar decompose, which can be seen by the change in colour of the core specimens in Fig. 11. The coupon manufactured at 190 °C for 60 min was unable to be tested, due to cracks caused by solidification shrinkage. The surface of this specimen was also tacky from the heavily caramelised binder.

From all of the mechanical test data available, it can be concluded that 150 °C is an optimal processing temperature for the current study. The trehalose binder is amorphous at this temperature, producing a dense core body with high mechanical properties that can resist resin penetration. However, in order to achieve further improvements in mechanical properties for cores manufactured at 150 °C, the processing time could be extended to 1 h without causing property degradation.

Table 4 compares the strengths of the NT Core (processing pressure 170 bar, binder content 8 wt%, processing temperature 150 °C, and processing time 60 min) and two commercial core materials at both

20 °C and 120 °C. Although the strengths of all materials drop at elevated temperatures, the NT Core has higher flexural and compressive properties compared to both benchmark materials, making it a potential alternative for industrial applications.

5.2. Post moulding operations

The geometry of moulded cores is limited by the non-hydrostatic pressure generated during the compression moulding process, as the salt/sugar mix does not flow when pressed. Large variations in mechanical properties can occur in complex cores due to different local compaction ratios. One solution is to cast a large block and then machine it to the final required geometry. This also creates opportunities for using this water-soluble core for lower volume applications, where the high cost of capital equipment and tooling cannot be amortised over the lifetime of the component. Trials have been conducted to demonstrate the feasibility of machining cast blocks of the NT Core material using two types of cutter: a ball-nose and an end mill. Only one configuration of the NT Core material was tested during this study, which was the configuration that gave the highest mechanical properties in Section 4.1. (The processing parameters for producing the NT Cores for

Fig. 9. Compressive strengths of NT Cores manufactured using different temperatures and times (different line colours) and tested at 20 $^{\circ}$ C (solid line) and 120 $^{\circ}$ C (dotted line).





Fig. 10. Flexural strengths of NT Cores made at various temperatures and times (different line colours) and tested at 20 °C (solid line), 80 °C (dashed line) and 120 °C (dotted line).



Fig. 11. Different colours of NT Cores made at various temperatures for different time.

Table 4

Comparison of strengths of the NaCl based core (NT) and other widely-used core materials.

| | Test temperatures | NT Core | A Core | B Core |
|---|------------------------------------|---|--|--|
| Compressive strength (MPa) Flexural strength (MPa) | 20 °C 120 °C 20 °C 120 °C | $57.3 \pm 1.8 \\ 19.7 \pm 0.9 \\ 10.2 \pm 0.6 \\ 3.2 \pm 0.2$ | $\begin{array}{rrrr} 1.6 \ \pm \ 0.1 \\ 0.6 \ \pm \ 0.1 \\ 2.8 \ \pm \ 0.1 \\ 1.4 \ \pm \ 0.1 \end{array}$ | $5.1 \pm 0.2 \\ 4.6 \pm 0.3 \\ 4.8 \pm 0.3 \\ 3.6 \pm 0.3$ |

Table 5

Roughness Ra at 6 locations (1 – Top surface; 2 – Cutting edge/Ball-nose cutter; 3 – Cutting surface/Ball-nose cutter; 4 – Cutting edge/Flat cutter; 5 – Cutting surface/Flat cutter; 6 – Inner cutting edge/Flat cutter).

| Location | 1 | 2 | 3 | 4 | 5 | 6 |
|----------|------|------|------|--------|------|------|
| Ra (µm) | 8.19 | 9.08 | 4.99 | 108.62 | 5.81 | 2.91 |



Fig. 12. The NT Core after end-milling with a Ø10 mm ball-nose cutter (a), a Ø10 mm flat cutter (b).



Fig. 13. The change in volume of B Core and NT Core (8 wt % binder content, pressed at 150 °C and 170 bar for 30 min) during water jet trials.

Fig. 14. An open-ended integrally-stiffened prepreg panel (a) NT Core in position and (b) after NT Core removal, showing blind hole.



this study were; pressure 170 bar, binder content 8 wt%, temperature 150 °C and processing time 30 min).

Fig. 12(a) shows that chipping was evident after milling the NT Core with a flat end mill. Damage occurred on the upper cut edges (Fig. 12(b)), which is indicated by the surface roughness value, Ra, in Table 5. The Ra values taken at Position 4 (which corresponds to the corner between the cut face and moulded face as shown in Fig. 2), are the only ones that significantly differ to the Ra values taken from the asmoulded surface (Position 1) – ~108 μ m VS ~8 μ m. Damage at this location occurs only during the first few cuts, as the flat end mill has a fixed and relatively small rake angle, which causes high cutting forces to be applied to the top surface of the core [22]. Chips therefore occur since there is no support for the surrounding material. However, cuts tend to become cleaner as the depth of the end-mill cutter increases, as indicated by the surface roughness measurements at Position 6 in Fig. 12(b).

Conversely, a clean cut can be produced with a ball-nosed cutter of a similar diameter, with no chipping during breakout. The contact area of the ball-nose cutter is initially lower and the rake angle increases gradually with increasing cutter depth. Therefore for the same spindle speed, there is less cutting torque applied on the cutting edge and the cutting force increases more gradually for subsequent cuts as the contact areas increases. More in-depth machining trials are required to be more conclusive, but this initial study has demonstrated the feasibility of machining large cast billets for producing complex geometries if casting net-shaped cores is not cost effective for low production volumes.

Post composite moulding core removal trials have also been conducted to compare the removal rate of the NT Core material with that of the commercial B Core. Fig. 13 compares core removal rates for the NT Core and the B Core, using a water jet at 8 bar and 40 °C. The B Core has a faster removal rate than the NT Core (fully removed after 15 s compared to 30 s) due to its low strength (5.1 MPa for the B Core versus 49.0 MPa for the NT Core). For the case shown in Fig. 14(b), the 50.1 g NT Core (27 cm³) was fully removed from the blind hole (open area of 338 mm²) in approximately 30 s, hence, the average removal rate was 54 cm³/min or 100.2 g/min, leading to a specific removal rate of 296 kg/(min·m²). The pressurised water therefore significantly improves the removal speed of the core compared with the SDR recorded by the water bath method (0.25 kg/(min·m²)), as the pressurised water provides some mechanical assistance to remove the core rather than solely relying on the dissolution of the salt and sugar.

6. Conclusion

A water-soluble NaCl-based core (NT Core) has been developed to enable complex hollow composite structures to be manufactured. Mechanical testing has shown that a compressive strength of 57.3 MPa and a flexural strength of 10.2 MPa can be achieved at ambient temperature. Although there is a clear reduction in mechanical properties at temperatures up to 120 °C, the results indicate that the core is suitable for high pressure moulding processes, such as compression moulding or HP-RTM, where in-mould pressures can exceed 100 bar. According to a statistical study, the mechanical performance of the core increases with increasing binder content, processing pressure, processing temperature, and processing time. However, the optimal processing temperature was found to be 150 °C, as the trehalose binder undergoes a phase change to a stable anhydrate and a state change to a liquid, enabling the binder to flow into the pores between the salt grains. Degradation of the binder was observed if the processing temperature was too high (190 °C) for long periods (over 30 min), significantly reducing the mechanical performance of the core.

Integrally-stiffened composite panels produced using the NT Core demonstrate that this material can be successfully removed by pressurised water through a blind hole, in order to produce hollow carbon fibre structures. Compared to other NaCl-based cores reported in the literature, the specific dissolution rate of the newly developed NT Core (170 bar and at 150 °C for 30 min, 8 wt% binder) is approximately 5 times faster (0.25 kg/(min·m²), since a water soluble binder has been used instead of relying on heavy mechanical interlocking of the salt grains. However, the overall removal rate (54 cm³/min) is around half that of the commercial B Core benchmark because of higher mechanical properties (flexural strength of 10 MPa vs. 4.8 MPa), since the main removal mechanism is fracture of the core by the kinetic energy of the water-jet, rather than dissolution. However, this is an inevitable compromise, as the superior mechanical performance is key to utilising this material for high pressure composite moulding processes.

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