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COMPOSITES THEORY AND PRACTICE

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EVALUATION OF VACUUM RESIN INFUSION PROCESS ON SELECTED GLASS FIBRE PREFORMS

The scope of the study is to evaluate the behaviour of exemplary glass fibre preforms during the vacuum infusion process. The reinforcing preforms were prepared from: plain weave crimp fabric, chopped strand mat (with two alternative binders - polyester-appropriate and universal), a unidirectional (UD) fabric and a 3 mm PARABEAM® 3D rising fabric. ESTROMAL 14 polyester resin was used as the matrix. All the laminates showed a fibre volume fraction in the range of 49÷52%. The permeability analysis was conducted with the use of PAM-RTM software by the ESI GROUP. As the base for the analysis, measured experimental saturation times were used. It is probably the simplest method to determine the permeability of fibrous preforms. The plain weave fabric showed a satisfactory saturation time (less than 9 min for 250 mm preform section) and permeability ($5.66 \cdot 10^{-10}$ m^2). The infusion process proceeded in a stable way and the resin front ran uniformly along the whole width of the preform. The chopped strand mat showed a saturation time similar to the plain weave fabric. However, it showed a very long time of saturation (over 46 min for 250 mm preform section) and low permeability ($1.06 \cdot 10^{-10}$ m^2) for the universal binder applied to the fibres. The universal binder probably does not react efficiently with terephthalate resins (ESTROMAL 14). The UD fabric showed evident anisotropy in permeability. When saturated along the main fibre strands, the permeability was by half higher than in the case of the plain weave fabric ($8.2 \cdot 10^{-10}$ m^2). When saturated transverse to the main fibre strands, it showed a permeability lower by about 40% in comparison to the direction along the main strands ($4.65 \cdot 10^{-10}$ m^2). The application of a spreading mesh considerably shortens the saturation time of the UD fabric (less than 1.5 min for 250 mm preform section), but it results in a deficient saturation. The PARABEAM® fabric showed a lack of the "rising" effect in the VIP process. However, further investigations may show some applications for this structure in pressure-assisted technological processes. The study showed that PAM-RTM software may be successfully applied to determine the permeability of fibrous preforms and to analyse the saturation processes. The determined values and trends in the K1 and saturation time are the initial assessment of preforms applicability in the vacuum infusion process.

Keywords: **laminate, preform, vacuum infusion, numerical simulation, rising fabric**

OCENA PRZEBIEGU PROCESU INFUZJI PRÓŻNIOWEJ NA WYBRANYCH PREFORMACH Z WŁÓKNA SZKLANEGO

Celem pracy jest ocena procesu infuzji próżniowej na wybranych preformach włókien szklanych, z zastosowaniem żywicy poliestrowej. Preformy zostały przygotowane dla: tkaniny rowingowej płóciennej (krzyżowej), maty (z dwoma alternatywnymi rodzajami lepiszcza - dla poliestrów oraz uniwersalnym), tkaniny jednokierunkowej (UD) oraz tkaniny wstającej 3D PARABEAM® o grubości 3 mm. Do nasycania użyto żywicy poliestrowej ESTROMAL 14. Wytworzone laminaty cechowały się udziałem objętościowym włókien w zakresie 49÷52%. Dla założonego programu eksperymentów przeprowadzono analizę przepuszczalności preform z użyciem oprogramowania PAM-RTM firmy ESI GROUP. Jako podstawę do tej analizy wykorzystano czasy nasycania zmierzone wcześniej w rzeczywistych warunkach laboratoryjnych. Jest to prawdopodobnie najprostsza metoda wyznaczania przepuszczalności preform włóknistych. Preforma płóciennej tkaniny rowingowej wykazała zadowalająco krótki czas nasycania (poniżej 9 min dla 250 mm odcinka) w procesie infuzji i dobrą przepuszczalność ($5,66 \cdot 10^{-10}$ m^2). Proces postępował stabilnie, a front żywicy przemieszczał się równomiernie wzdłuż całej szerokości preformy. Mata z lepiszczem dla poliestrów wykazała podobny do tkaniny krzyżowej czas nasycania oraz przepuszczalność. W przypadku preformy maty z lepiszczem uniwersalnym zaobserwowano bardzo długi czas nasycania (ponad 46 min dla odcinka 250 mm) i niską przepuszczalność ($1,06 \cdot 10^{-10}$ m^2). Tkanina UD wykazała ewidentną anizotropię w przepuszczalności. Przy nasycaniu w kierunku zgodnym z ułożeniem pasm włókien jest ona o połowę wyższa niż dla tkaniny krzyżowej ($8,2 \cdot 10^{-10}$ m^2). Przy nasycaniu w kierunku poprzecznym do pasm włókien przepuszczalność jest o ok. 40% niższa niż dla kierunku wzdłuż pasm ($4,65 \cdot 10^{-10}$ m^2). Nałożenie na preformę siatki rozprowadzającej znacznie skraca czas nasycania tkaniny UD (poniżej 1,5 min dla odcinka 250 mm), ale skutkuje niedosyceniem przekroju preformy - od 1 do 3 warstw pozostaje częściowo nienasyconych. Tkanina PARABEAM® nie wykazała efektu „wstawania" podczas procesu infuzji z powodu docisku worka. Jednakże, prawdopodobne jest wykazanie w dalszych badaniach ewentualnej przydatności tej tkaniny dla procesów ciśnieniowych. Niniejsze studium wskazuje, że program PAM-RTM może być z powodzeniem zastosowany do wyznaczania przepuszczalności preform i do analizy procesów nasycania. Wyznaczone wartości i trendy K1 oraz czasu nasycania stanowią wstępne szacowanie przydatności preform dla procesu infuzji.

Słowa kluczowe: **laminat, preforma, infuzja próżniowa, symulacja numeryczna, tkanina wstająca**

INTRODUCTION

The vacuum infusion process (VIP) has recently become one of most popular methods for manufacturing FRP (fibre reinforced polymer) laminates [1-3]. It is a modern method, advantageous especially from: the *industrial safety*, *environmental protection* and *productivity* points of view [4-6]. Vacuum infusion is a very universal method. It may be applied in most product types - among others in parts for automotive [6], naval [5], aerospace [7] solutions as well as in containers and pipes [8, 9]. It is also suitable for all types of 3D preforms, among others for stitched laminates [10, 11]. As confirmed by the authors of the study in previous investigations, vacuum infusion is a very good method for manufacturing products from natural fibre reinforced polymer (NFRP) composites [12, 13]. However, the VIP method is sometimes problematic, especially for complicated shapes of products (like fan blades). Each application demands a very precise process design. The design procedure consists in optimizing the arrangement of inlets and vents and with a proper choice of process parameters. The main process parameters are: temperature (determining the rate of resin curing), resin viscosity and applied pressure gradient (determining the rate of resin flaw). Additional variables concerning the applied materials are: resin homogeneity, resin thermal conductivity and permeability of the preform (fundamental). The initial design of the process parameters and materials (resin and preform) properties is performed for various purposes: to "steer" the process, to shorten the process time and to improve the quality of the produced laminates. Attempts were made to modify the resins in order to make them more "controllable" and predictable during the process, exemplary by the addition of mineral fillers [14], carbon nanotubes [15] or metal (copper, aluminium) powders and flakes [16, 17]. Moreover, chemical modifications of the resins seem promising - for example photoactive resins [18] or liquid-crystal epoxy resins [19]. Numerous studies have been conducted on VIP technology, especially regarding the influence of the parameters and materials variables on the process efficiency and quality of the manufactured product - exemplary studies are [12, 20, 21]. The application of computer simulations performed on dedicated software (among others PAM-RTM, RTM-WORX) is a very important part of the VIP process design. They enable one to design an arrangement of inlets and vents along the preform - it is indispensable for big or/and complicated elements. The preliminary experimental determination of preform permeability is always necessary to perform the simulation.

The paper presents an attempt to evaluate how the structure of the following model fibrous preforms behave during the vacuum infusion process: 10 layers of plain weave fabric, 6 layers of chopped mat, 6 layers of unidirectional (UD) fabric, 3 mm thick PARABEAM® 3D rising fabric. The evaluation contained a measurement of the saturation time, visual observation of the process progress and visual evaluation of the cured laminate panels. The results of the experimental measurements of saturation time were used to perform a simulation of the vacuum infusion process, with the use of PAM-RTM software, during which the permeability of the tested preforms was determined. Such an approach is very interesting and it is probably the simplest method to determine the permeability of preforms for FRP composites. PAM-RTM by the ESI GROUP is a finite element based software dedicated to simulate the processes of composites saturation - RTM (resin transfer moulding), vacuum infusion, RFI (resin film infusion). Precise information about the software is available on the web page of the general Polish representative of the ESI GROUP [22].

The results and conclusions obtained within the study will be used in the design of the VIP process for composite fan blades.

MATERIALS AND METHODS

Glass fibre - polyester resin laminates were manufactured by the vacuum resin infusion method. The reinforcing preforms were prepared using the following model glass fibre structures: ***1)*** KROSGLASS ***plain weave crimp fabric*** (equal fibre volume into weft and warp directions) 350 g/m^2, based on ER 3003 roving, 10 layers, 0/90 lay-up; total areal weight of the preform 3500 g/m^2, ***2)*** KROSGLASS ***chopped strand mat*** 560 g/m^2, based on ER 3003 roving, 6 layers; total areal weight of the preform 3360 g/m^2; the mats with two different binders were used alternatively: *polyester-appropriate binder* and *universal binder*, ***3)*** KROSGLASS ***unidirectional (UD) fabric*** 620 g/m^2, based on ER 3003 roving, 98.5% of fibres in the weft direction, 1.5% of fibres in the warp direction, 6 layers, unidirectional lay-up; total areal weight of the preform 3720 g/m^2; spreading mesh (nylon, 105 g/m^2, about 1 mm in thickness) was applied on the top of the lay-up for part of the preforms to improve the spread rate of the resin, ***4)*** PARABEAM® 3 mm ***3D rising fabric***, total areal weight 780 g/m^2. All the preforms were to have an areal weight possibly close to 3500 g/m^2 (with exception of PARABEAM® fabric) and a thickness of about 3 mm. These dimensions equal the preform of a 10 layer thick lay-up of 350 g/m^2 plain weave glass fabric, which was researched by the authors in a number of earlier studies [9-13].

The system: ESTROMAL 14 terephthalate polyester resin by ERG PUSTKOW (low styrene emission, density at 1.14 g/cm^3 when cured) +1.5% by weight of BUTANOX M-50 (methylethylketone peroxide dissolved in dibutyl phthalate; contains more than 10% active oxygen) hardener, was applied as the matrix in all the studied laminates. These proportions guaranteed the time to gelation initiation in the range of 50÷60 min.

The viscosity of the resin + hardener system was determined by the Ford cups method and averages about 1000 mPa·s (1 Pa·s). The vacuum infusion process was conducted on the system presented in Figure 1. The mold had the form of a flat plate. The spreading spirals ensured steady distribution of the liquid resin flow along the width of the saturated preform. Therefore, the resin flow had only a unidirectional character.

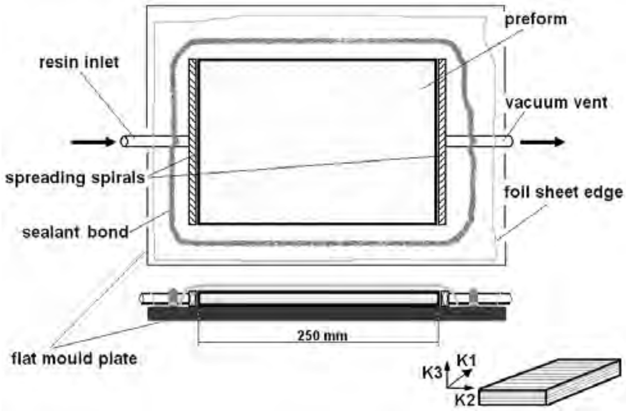

Fig. 1. Resin vacuum infusion system applied for panels manufacturing. Permeability tensor components directions are marked on ideal scheme of preform

Rys. 1. Układ infuzji próżniowej zastosowany do wytworzenia płyt. Na schemacie ideowym preformy zaznaczono kierunki składowych tensora przesączalności

The differential pressure between the vent and the inlet (resin front) was about 283 hPa (–0,72 bar on the manometer) for all the cases. The process was conducted at a constant temperature of 22°C and ambient pressure of 1010 hPa. The resin flow lasted to the moment the whole preform was filled and the vacuum conditions were maintained to the moment the laminate was initially cured.

Saturated laminate panels were cured at room temperature - about 22°C - for 24 h (including gelation under vacuum conditions) and additionally after-baked at 55°C for 6 h. All the laminates had a fibre volume fraction in the range of 49÷52%. It was determined by comparison of the mass of the "dry" preform and of the same preform after saturation, the curing process and removal of the matrix allowances (gravimetric method). The saturation time measurements were conducted for all the specimens. It consisted in marking the lines the flowing resin front achieved in a particular period of time (usually after: 1, 2, 3, 5, 10, 15, 20 min and every next 10 min if needed). The distances between the line of the first-exposed preform edge and each marked line, corresponding to the proper time, gave information about the saturation rate. The measured saturation times are presented in Table 1.

Permeability (k) is a measure of the ability of a porous material (fibrous preform) to allow fluids to pass through it [23]. For the unidirectional flow of fluid through homogeneous porous media it is defined by the following formula:

$$k = v \cdot \frac{\mu \cdot \Delta x}{\Delta P} \tag{1}$$

where: k is the permeability [m^2], v is the superficial fluid flow velocity [m/s], μ is the dynamic viscosity of the fluid [Pa·s], Δx is the thickness of the bed of the porous medium [m], ΔP is the applied pressure difference [Pa] [23].

TABLE 1. **Results of experimental saturation time evaluation and numerical analysis of permeability for tested preforms**

TABELA 1. **Wyniki eksperymentalnej oceny czasu nasycania oraz przesączalności wyznaczonej podczas analizy numerycznej dla badanych preform**

Preform type	Saturation time of 250 mm preform section [s]	Permeability K1, 10^{-10} [m^2]
Plain weave fabric 3500 (10 layers)	526	5.66
Chopped strand mat 3360 (6 layers) - polyester-appropriate binder	574	5.19
Chopped strand mat 3360 (6 layers) - universal binder	2782	1.06
UD fabric 3720 (6 layers) - resin flow along weft strands	361	8.2
UD fabric 3720 (6 layers) - resin flow transverse to weft strands	630	4.65
UD fabric 3720 (6 layers) - along strands + spreading mesh	88	34.4
PARABEAM 3 mm (along "channels")	313	9.58

In anisotropic media the permeability is a tensor quantity. In the applied unidirectional saturation system (Fig.1), only the component corresponding to the resin flow direction (designated as K1) is important. Permeability is a material property of a fibrous preform and it is the objective quantity to analyze the behaviour of the material in technological conditions. The permeability analysis was conducted with the use of PAM-RTM software by the ESI GROUP. As the basis for the analysis, the measured experimental saturation times were used - they were put into the program and it calculated the permeability. It is probably the simplest method to determine the permeability of fibrous preforms. The constant parameters: pressure (283 hPa) and resin viscosity (1000 mPa·s), were also put into the program as necessary data for permeability analysis. The determined K1 values are presented in Table 1.

EVALUATION OF THE RESULTS

The plain weave crimp fabric showed a satisfactory saturation time and good permeability (Table 1). The time of 10 min for 250 mm of preform is very reasonable for such a type of structure, by applying an average-performance vacuum pump (above –0,95 bar). It is comparable to industrial achievements [5, 9, 10, 12, 24]. The process proceeded in a stable way and the resin front ran uniformly along the whole width of the preform. Organoleptic evaluation was performed to check the quality of the cured laminates. The quality of the plain weave laminate was very good - the panel showed uniform, undisturbed transparency over the entire surface. The average fibre volume fraction was 51.7%. The chopped strand mat showed saturation behavior similar to that of the plain weave fabric (Table 1). The quality of the manufactured laminate was also good. The average fibre volume fraction was 49%. Good filling of the mat and good wetting (clear transparency) is visible in Figure 2a.

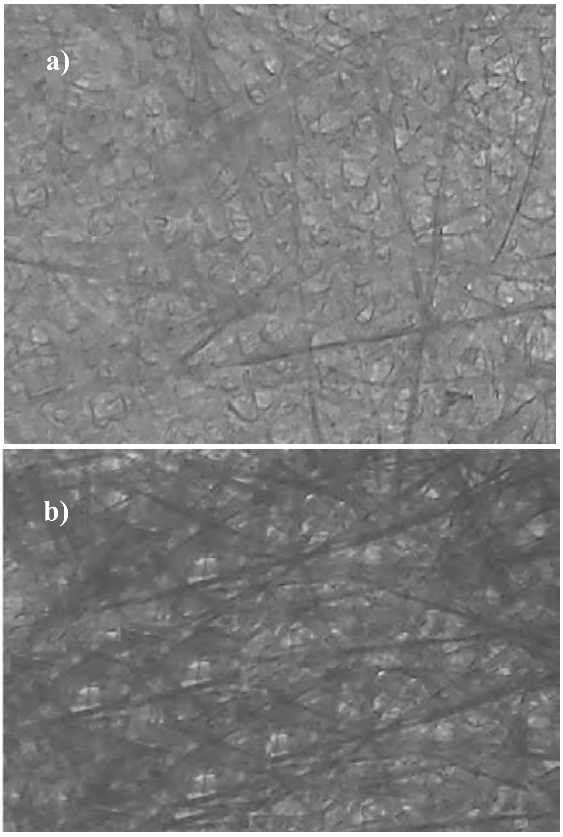

Fig. 2. Structure of chopped strand mat after saturation by vacuum infusion: a) properly saturated mat, b) poorly saturated mat - covered with "universal" end-use finish

Rys. 2. Struktura maty po nasyceniu metodą infuzji próżniowej: a) właściwie nasycona mata, b) źle nasycona mata - pokryta „uniwersalną" apreturą

The chopped strand mat with the alternative binder showed a very long time of saturation and low permeability (Table 1). It probably arose from the inferior properties of the end-use finish, because the rest of the parameters were not changed. According to the notations of the technical specifications, it is a universal binder (for polyester, epoxy and vinylester resins), but it evidently does not improve the wetting of the fibres with the applied terephthalate polyester resin (ESTROMAL 14). The quality of the manufactured laminate was inferior. Poor filling of the fibres and lack of wetting is well visible in Figure 2b. The strand mat with the "universal" binder is obviously the hardest structure to saturate from among the studied preforms. Probably, the mat with the "universal" binder could be used for a hand lay-up manufacturing method where saturation of the fabric may be "forced" by the strong action of a brush or roller.

The UD fabric showed evident anisotropy in permeability (Table 1). When saturated along the fibre strands, the permeability was by half higher than in the case of the plain weave fabric. When saturated transverse to the fibre strands it showed a permeability lower by about 40% in comparison to the direction along the strands. The quality (by organoleptic valuation) of the manufactured laminate was good. The average fibre volume fraction was 49.8%.

The spreading mesh applied on the top of the lay-up shortens the saturation time (Table 1). However, it results in a lack of saturation in 1-3 layers within most of the laminate area (Fig. 3).

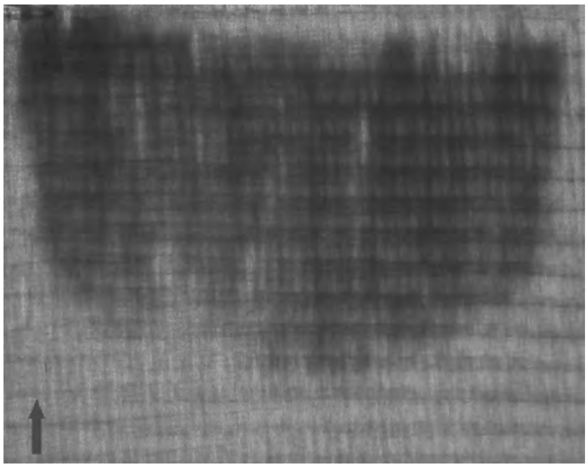

Fig. 3. Cured UD laminate panel after infusion process with spreading mesh. Resin flow direction has been marked

Rys. 3. Utwardzona płyta laminatu UD po procesie infuzji z siatką rozprowadzającą. Zaznaczono kierunek przepływu żywicy

According to the producer's guidelines, PARABEAM 3D rising fabric is suitable only for hand lay-up technology. However, it is a very interesting structure and it could play a valuable role in numerous advanced constructions achieved by RTM or vacuum infusion methods. It also could be a very good material for the stiff walls of industrial fan blades. Therefore, the authors incorporated PARABEAM to the study.

The fabric occurred to be unsuitable for vacuum infusion technology. After being wetted with the resin, the two-face layers of a rising fabric should be joined by a row of transverse "walls" risen by wetting forces (Fig. 4a). During the infusion process, the layers are pressed by a vacuum bag and the "walls" cannot rise. It results in a thin 2-layer structure (Fig. 4b) devoid of high stiffness, being the main advantage of rising fabrics.

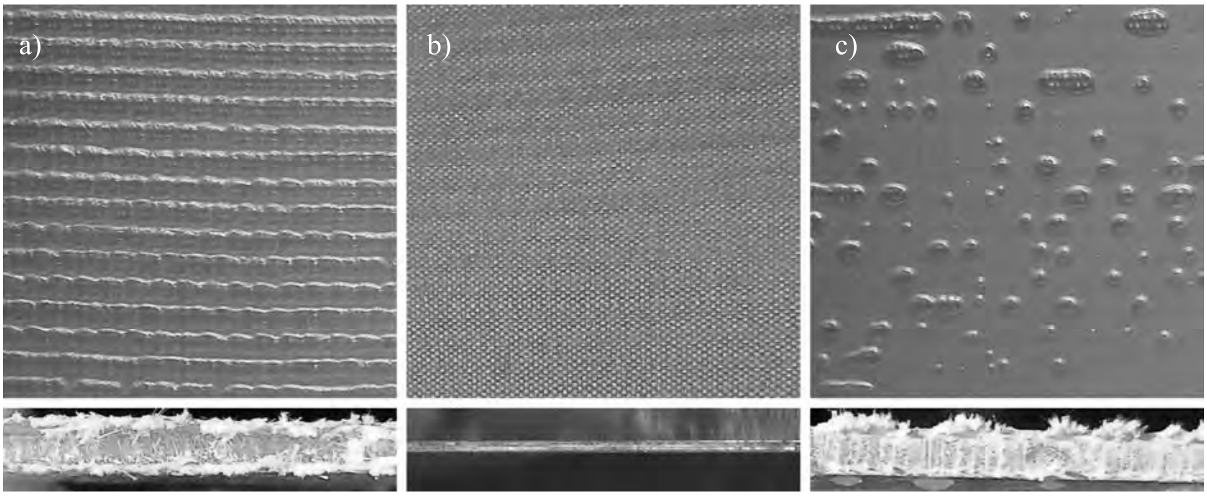

Fig. 4. Structure of PARABEAM standing fabric (3 mm) after saturation by various methods: a) hand lay-up (according to producer's guidelines), b) vacuum infusion, c) RTM

Rys. 4. Struktura tkaniny wstającej PARABEAM (3 mm) po nasyceniu różnymi metodami: a) kontaktowo (zgodnie z wytycznymi producenta), b) infuzja próżniowa, c) RTM

The RTM (in stiff mold) process in laboratory conditions was also applied especially for the PARABEAM saturation - a mould with a 3 mm thick nest was used. It resulted in filling the "channels" between the "walls" with resin (Fig. 4c). In the applied conditions there was no possibility to pump or drift the excess resin. Such an over-filled structure is heavy and unusable.

Only the hand lay-up method with the use of a strict dose of resin guarantees the proper structure of PARABEAM (risen "walls", empty "channels" - Fig. 4a). However, further technological tests may show the applicability of PARABEAM in vacuum and RTM methods. It would demand applying some extraordinary procedures - for example: shutting off the vacuum after saturation by the vacuum infusion process, pumping or gravitational drift of excess resin after the RTM process.

The study showed that PAM-RTM software may be successfully applied to determine the permeability of fibrous preforms and to analyse the saturation processes. The determined values and trends in K1 and saturation time are the initial assessment of the preforms applicability in the vacuum infusion process.

CONCLUSIONS

The tested preforms behaved in the vacuum infusion process as follows:

1. Plain weave crimp fabric showed satisfactory saturation time and permeability during vacuum infusion. Process proceeded in a stable way and resin front ran uniformly along whole width of preform.
2. Chopped strand mat showed saturation time similar to plain weave fabric.
3. Chopped strand mat showed very long time of saturation and low permeability when improper binder was employed.
4. UD fabric showed evident anisotropy in permeability. When saturated along fibre strands, permeability was by half higher than in the case of plain weave fabric. When saturated transverse to fibre strands it showed permeability lower by about 40% in comparison to direction along strands.
5. Spreading mesh shortens saturation time of UD fabric, but results in deficient saturation.
6. PARABEAM® fabric is not suitable for vacuum infusion technology. However, further investigations may show some applications for this structure in pressure-assisted technological processes.
7. PAM-RTM software may be successfully applied to determine permeability of fibrous preforms and to analyse saturation processes.

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