MANUFACTURE AND TESTING OF 3D WOVEN NATURAL FIBRE COMPOSITES

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SUMMARY

This paper reports new developments in the design, manufacture and testing of 3D reinforced natural fibre woven composites with structural strength potential. It discusses the 3D weave architectures used, details the VARTM composite processing method and highlights the challenges of producing structural thermoset composites from naturally variable yarns.

Keywords: 3D woven, natural fibre, fibre reinforced composites, VARTM

3D WOVEN COMPOSITES RESEARCH

Driven from aerospace and military research, optimum 3D reinforced super-materials have emerged within the composites sector. The high density specifications in terms of preform and composite fibre volume, exact yarn placement and target production quantities of new aerospace composites have fuelled the need for specialist customisation of loom technology, Jacquard shedding mechanisms and weft insertion methods. Unique weave manufacturing devices, multi-insertion yarn systems, multi-directional reinforcement and customised shaped structures in high performance fibres with extreme specifications and tolerances have been developed, mainly in the USA, UK and Germany [1,2,3,4,5,6]. Indeed, narrow fabric weaving, pile weaving and heavy industrial belt-weaving have all influenced developments to see ‘application specific’ set-ups, where one unique machine is set-up for one type of fabric.

With the anticipated growth and broadening interest in these materials, the 3D woven composites sector needs to demonstrate its scope in terms of providing materials with tailored properties and superior strength to weight attributes at a competitive cost. The expertise and technical underpinning that supports these niche composite materials can therefore be redirected and utilised in lower specification, load-bearing textile composite applications targeted at less stringent non-aerospace domains. This new research highlights how multiple layer 3D woven composites are being reconfigured for potential use in transport, marine and infrastructural applications using naturally derived fibres such as flax and viscose rayon, using conventional based weave technology.

With additional advantages of renewable supply, near-net shaping and future capability for volume production, cellulose based composites position themselves as a major contributor to future composite applications. Examples include generation of composite specimens for generation of mechanical property data, near-net shaped prototypes for transport /performance automotive, thick reinforcement panel prototypes, and structural reinforcements for building and infrastructure.

THE DRIVE FOR NATURAL FIBRE COMPOSITES

With the implementation of EU directives [7] on waste and end of life disposal strategies, there has been a surge of activity driven by the European Automotive Industry to introduce Natural Fibre Composites (NFC) as a substitute to glass fibre (GRP), where appropriate. The production of these non-woven and plied single layer laminates has significantly escalated within the last 5 years due to demand from the automotive industry and the need for industries to develop by-products from their waste materials in response to directives to further reduce the accumulation of waste into landfill sites.

NFC materials derived from cellulose-based matter primarily include agricultural waste from a variety of crops, chopped strand mats or single woven plies, which are mixed with an adhesive matrix and compression moulded to form fibre panels and plied laminate structures.

The ability to thermally recycle NFC's (depending on resin matrix), and reduce vehicle weight thus reducing fuel consumption and CO_2 emissions makes natural fibre composites a desirable commodity for the future. With sustainable, lower environmental production impact and lightweight attributes, matched with low cost in comparison to glass fibres as driving factors [8,9], natural fibre nonwovens have desirable characteristics for use in interior trim and non-critical vehicle parts. Material data is continually being generated allowing greater usage and integration into further applications. By addressing the technical challenges, NFC's will be utilised in structural applications. Goutianos [10] concluded that in order to enable the use of natural fibre composites in high-quality engineering applications, the development of unidirectional tapes, and non-crimp and woven textiles was required. These types of natural fibre systems could then be more easily integrated into traditional large scale industrial composite processing techniques. Using higher specification, more costly woven or knitted technologies require investment in order to establish their mechanical and economic crudentials before being accepted for use.

The restriction in increased uptake of NFC's thus far, has been due to the natural variability and inconsistency of the materials, the limited mechanical performance of non-woven and plied lay-up preform assemblies, the commercial availability and proven performance of a naturally derived bioresin to form 100% recyclable composites, and cost. The expenses associated with raw materials and components, the material weight and the ability of the material to be processed using existing technology are also important factors to consider [9].

Substantial research to address these key areas is currently underway and significant insights have been reported, particularly in the treatment of natural fibres for improved fibre-matrix adhesion [11,12], the development of naturally derived resins from plant oils [13], performance of UD and laminated natural fibre composites [14] and economic research [15]. Preforms used by the leaders in this field [11-17] mainly use non-woven fibre mat products in flax, hemp, and hybrid mixed fibre assemblies with uni-directional or orientated fibre direction, or single layer woven plies that rely on the reinforcement-matrix interfacial strength targeting appropriate yet non-critical load-bearing applications. Multiple plies of fabric form stacked laminates to obtain desired thickness (2-10mm) and strength and some can be reinforced further using robotic stitching methods. Flax yarn producers are experiencing a renewed demand for high quality yarns and fabric manufacturers who are equipped with the necessary technology are introducing new heavier weights of biaxial woven fabric designed specifically for use in NFC laminates [18].

However the lack of continuous fibre integrity within the textile part is a significant factor which inhibits their selection for higher specification structural parts with load-bearing capabilities. Scobie [19] states that 'lignocellulosic fiber resin composites have suffered from lack of strength because the resin and fiber have been combined without the structured fiber formation.'

Data related to NFC's using 3D woven or 3D knitted spacer preform reinforcements [20] in natural fibres is limited as fabricators specialising in these fabric types have been mainly funded to conduct aerospace or biomaterials research. In response to these issues, this work aims to concentrate on 3D reinforced woven NFC's that will provide continuous fibrous reinforcement in the three principle stress directions.

NATURALLY-DERIVED FIBRES AND YARNS

Non-woven mats can be made from low-grade decorticated bast fibre or refined shive [13]. No refining or spinning is required. For weaving, the sliver must be high quality and refined through numerous combings to produce a spun yarn sturdy enough for fabric manufacture [21]. Sourcing the optimum heavy count yarns has posed several challenges in terms of negotiating between quality, performance and cost. The grade of raw material from the field varies between batch yield and country of origin. Cost for flax yarn ranges from Euro 2 – 10/kg. Cheaper flax yields exhibit poor fibre quality and are subject to increased fibrillation damage and fibre degradation during the weave process. Therefore, a high quality variety must be used.

Natural fibres are subject to treatment preparation, shrinkage and non-uniformity in comparison to glass or carbon fibres. It is important that yarn manufacturers develop appropriate finishing methods for yarns that can then be converted into fabric for NFC materials, and it is expected that this will be reflected by an escalation in production cost. Carbon and glass fibre yarn manufacturers have been developing and optimising the sizing agent and compatible chemical treatments for enhanced adherence between fibre and resin matrix. Since woven NFC's are in their infancy, it is only to be expected that compatible sizing and finishing treatments applied to weaving yarns are yet to focus on the adherence of fibre to resin matrix, and thus requires further research.

This research has also considered the use of fibres naturally-derived, yet synthetically produced. Viscose rayon yarns produced from wood pulp have been synthetically modified by the manufacturer to produce consistent properties which increases its attractiveness for engineering composite applications. The fibre does not require treatment and achieves good thermal and adhesion compatibility with the resin matrix. Attributes such as consistency in production and fibre properties, small batch supply, comparable cost and quality certification, demonstrated the potential as a yarn to be investigated in this research.

The pretreatment of natural fibres is recommended to improve fibre /matrix interfacial adhesion [11,12]. Alkyl, saline or enzyme treatments can be used. However, the benefits of fibre treatment come at a price in terms of the decrease in mechanical properties. Comparisons were made on the inherent untreated properties of flax yarns treated with 4% sodium hydroxide (NaOH). Flax yarns (400 tex), both treated and untreated were prepared with a 150mm gauge length, tabbed with 3 ply 25mmx35mm cardboard tabs and subjected to tensile testing according to BS 10618. It was determined that the treatment with sodium hydroxide had a significant negative effect on the tensile properties of the flax yarns. Drops of 73% and 76 % in strength and modulus were evident in the virgin yarns. Impregnated yarns sustained an 11% drop in strength but no change in the modulus. Consequently, the flax preforms processed into composites discussed later were not treated with this process. However, the treatment can lead to improved composite properties as the alkyl solution induces removal of pectin and lignin in the natural fibre, increasing aspect ratio, which can be advantageous to composite performance.

Flax Yarns	Virgin		Impregnated	
	Untreated	Treated	Untreated	Treated
Tensile Strength (MPa)	82.38	20.93	71.34	62.55
Tensile Modulus (GPa)	5.0	1.2	5.4	5.4

Table 1: Results of Flax Yarns Untreated/Treated

WEAVE PRODUCTION

Automated multi-shaft Dobby and electronic Jacquard looms at University of Ulster facilitates normal and complex set-up operations to fabricate small sample batches. This adaptable approach prevents the expense of ‘down-time’ that is incurred whilst reconfiguring a production volume machine in industry. Although slower in production speed, the time spent weaving can be off-set to a degree by the time-saved in set-up costs. In order to maximise output and range, several fabric types can be woven with the same set-up on a multi-architecture sampling facility. Near-net shaped architectures normally require a uniquely configured set-up.

Weft insertion methods include rapier and automatic shuttle methods (which produce a natural selvedge). Loom parts such as heddles and beat-up reeds often contribute to fibrillation damage of warp yarns and can restrict the alignment and positioning of warp yarns in the preform. Modification to moving parts and customised components can be introduced to minimise fibre damage, enabling production speeds to be increased. The preparation of warp beams and creels requires warp winding facilities and often rewinding of packages to fit specific creels. This additional overhead needs to be accounted for in the overall set-up costs. Fibre waste accumulated during fabrication can be redirected for use in non-woven by-product research, such as investigating life cycle assessment.

A generation of CAD design systems have evolved to simulate and aid in the design of 3D wovens. The idealised simulation of the architecture gives a good indication of yarn paths but mapping the distortion that arises during fabrication is more complex as it varies with each architecture, production settings and yarn type used.

3D WEAVE ARCHITECTURES

A loom will have several optimum set-ups to enable small sample batch production of integrated, orthogonal, angle interlock and near-net-shaped / tailored preform ranges. Integrated reinforcements are characterised by using plain, twills and satin weaves to form preforms with characteristics that reflect the inherent nature of the traditional weave assigned in the structure. (E.g. satins display good drapability with uniform surfaces, plains display high crimp etc.). Orthogonals primarily contain straight stuffer yarns with fine binder yarns penetrating the preform thickness and containing very high warp and weft sett densities to obtain high areal density values. These are the desired architecture for aerospace applications. Near-net shaped preforms are custom designed assemblages that often contain unique warp paths and hybrid combinations of structure, yarn types and tailored properties into localised regions. Due to their bespoke nature, they are usually custom one-off product specific preforms, and are therefore costly and challenging to produce.

Soden [22] has developed extensive ranges from 3 to 20 layer structures with areal densities in the range 1000-3000 g/m^2 and up to 5000g/m^2. An ever-expanding range of application specific, thick, multiple layer 3D reinforced structures, and new innovative shaped configurations have been produced. Tailored towards more demanding structural components, these natural fibre materials aim to demonstrate enhanced mechanical properties compared with nonwoven, or plied woven laminates used currently.

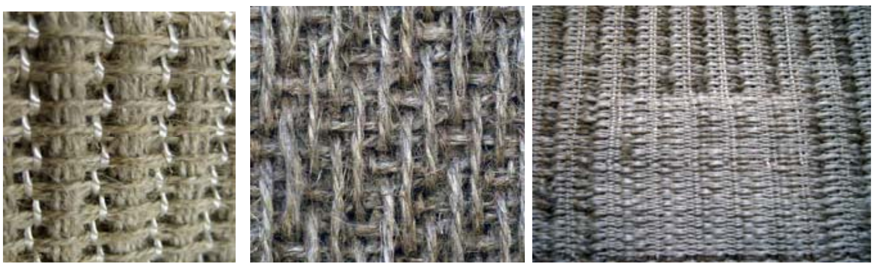

Fig 1: 3D woven natural fibre orthogonal (Left), integrated (Right)

and tailored hybrid preform (Bottom)

For this study, flax and viscose rayon angle interlock preforms have been selected. Warp yarns contribute both to the X-Axis and the Z-Axis reinforcement by migrating through the total thickness at an angle of approx 45^o one layer at a time. This forms a structure with equal yarn path lengths between layers and provides a uniform preform surface for consolidation in the mould tool.

As a consequence of designing to achieve lower areal density specification (1500-1800g/m^2), preform thickness is reduced, warp and weft sett densities are lower and this ultimately affects the inclination angle of the through-the-thickness or Z-orientation binder yarn. The inclination angle of these yarns are found to be in the range of 30^o – 60^o. Although contributing to the preform integrity and minimizing crack propagation between layers, research into this effect [23] has recommended that for improved mechanical performance, the inclination angle of the TTT binder should be as near to perpendicular (90^o) as possible.

Spun flax yarns are bulky and textured in nature which helps retain their cross-sectional shape and resist deformation in the preform. Viscose rayon yarns are slippy in nature and are prone to distortion and flattening to an elliptical shape within the preform. This is reflected in the varying preform thickness between the fabric types and is pronounced in the cross-sectional micrographs in Fig.3. Due to the slippage experienced with viscose yarns, additional tension is applied during manufacture and this can affect the weft pick density values.

Structure	**Ends/cm**	**Picks/cm**	**Thickness (mm)**	**g/m^2**
Flax interlock	22.4	17.2	5.5	1697.00
Viscose interlock	22.4	15.2	2.8	1505.00

Table 2: Preform details

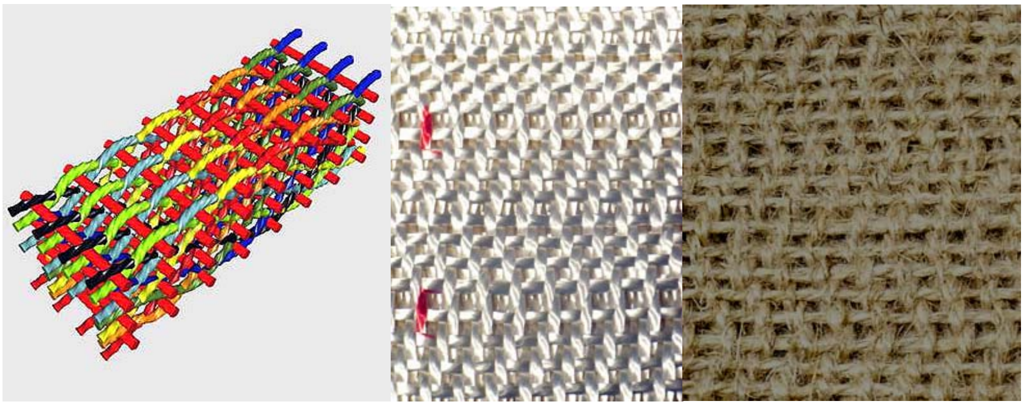

Fig 2: 3D Angle interlock design simulation (left), viscose preform (centre) and flax preform (right)

RESIN PROCESSING

Due to the challenging processing and performance characteristics desired in engineering composite applications, there are limited bio-resin systems commercially available which are completely sustainable in origin, but their inevitable development represents significant stages on the route to entirely biodegradable thermoset composites [9]. Introducing a sustainable reinforcement, such as the specimens reported in this research, contributes significantly in the quest to achieve a fully sustainable natural fibre composite.

A two-part epoxy resin system; Araldite LY-564 epoxide and an Aradur HY 2954 curing agent was used as the baseline resin matrix. Both epoxide and amine curing agent were supplied by Saint Gobain Vetrotex. A mixing ratio of 100:35 by weight of epoxy to curing agent was used as recommended by the manufacturer to create a stoichiometrically balanced epoxy resin. The epoxy was outgassed for 60 mins at 30°C prior to injection.

In addition to the standard epoxy system, a novel thermosetting bioresin; BioRez™ from TransFurans Chemicals bvba was also employed as a matrix material for an initial feasibility and comparative study. The furan resin, supplied as a one component system is based on furfuryl alcohol derived from Hemi-cellulose C5 sugars produced from agricultural waste.

Vacuum Assisted Resin Transfer Moulding (VARTM) was used to manufacture the composites. The viscose rayon and flax fabrics were cut into 350mm x 350mm specimens. No fabric treatment was undertaken prior to lamination. The dry preforms were placed in the mould, flexible tooling allowed 100 KPa pressure to compact the preform and the resin was injected into the mould under 75KPa at 75°C for the epoxy composites and 100KPa for the furan composites at 110°C. Wet out of the preforms occurred after 7.5 and 10.5 minutes respectively. After wet-out the viscose rayon/epoxy composite was ramped to 100°C at 0.64°C/min and held isothermal for 60 minutes. A post cure of 8hrs at 145°C was carried out on epoxy composites. The viscose rayon/furan composite were de-moulded 20mins after wet-out and B staged at 145°C for 60 min.

FLEXURAL TESTING AND V_F DETERMINATION

Flexural tests were carried out according to BS 14125 using a three point bend configuration with a span to thickness ratio of 25/1 and 10mm diameter load/support rollers. Five samples were tested per composite and all samples failed within 30-180 seconds.

Fibre volume fraction tests were carried using the density buoyancy technique on a Mettler Toledo XS64 at 17.4°C. A few drops of a wetting agent were added to distilled water and the density of the materials calculated via displacement according to ASTM D 792-00. At least three composite specimens were tested per structure and it was assumed that there were no voids in all composite samples tested.

RESULTS/DISCUSSION

An average V_f of 20% and 40% for the flax and viscose rayon composites respectively were calculated from the densities measured as shown in table 3. Difficulties were encountered measuring the flax yarn density. 100mm lengths of yarn were initially tested; however these produced an average density of 1130kg/m^3, a value approximating epoxy density. The yarn length was reduced to 10mm and the two-ply tows which make up the 400 tex yarn were separated and their twist removed. Half of these yarns were conditioned in a vacuum oven for 60 mins at 100°C and the other half tested as normal. Both the conditioned and unconditioned yarns produced an average density of 1560kg/m^3 which approximates other density values reported in the literature [14]. Additionally the composites V_f was crosschecked via preform mass and area measurements, final moulded thickness measurements and an assumed yarn density (1560 kg/m^3). A final composite V_f of 23% was calculated. This approximates the value found via the density measurement method. It should be noted therefore that the composite V_f for the flax laminate is not strictly exact.

Specimen	Density (kg/m^3)
Viscose rayon yarn	1461
Flax yarn	1560
Epoxy resin	1131
Viscose rayon/epoxy composite	1264
Flax/epoxy composite	1211

Table 3: Density results for composite materials

The flexural results of the viscose rayon composites are given in table 4. The viscose rayon/furan composites with a final moulded thickness of 2.38mm produced an encouraging flexural strength value of 168.5 MPa and modulus of 6.7 GPa.. The modulus equaled the viscose rayon/epoxy composites (2.38mm thickness) performance whilst a drop of 23% in flexural strength was observed. These 3D woven viscose rayon/furan values compare with data in the literature [24] for 2D glass/furan composites which report flexural properties of 104.6 MPa strength and modulus of 9 GPa with a 57% V_f, giving 73 MPa and 6.3 GPa if normalized to 40% V_f.

Composite	Average flexural strength (MPa)	s.d. (MPa)	s.d. (%)	Average flexural Modulus (GPa)	s.d. (GPa)	s.d. (%)
Viscose rayon/epoxy	168.5	10.1	16.6	6.7	0.5	13.0
Viscose rayon/furan	130.4	9.3	14.0	6.7	0.4	18.4
Flax/epoxy	68.8	6.1	11.2	4.6	0.3	16.5

Table 4: Flexural results on viscose rayon composites along the warp orientation

Both preforms experienced 15% compression during consolidation. Polished microsections were used to analyse the architecture of the textile composites along the warp orientation as shown in Fig. 3. The flax/epoxy composite yarns migrate through the thickness at 45° approx. displaying good alignment free from distortion, with the angled yarn paths of the interlock architecture clearly intact. This contrasts to the viscose rayon/epoxy architecture in which yarn slippage, high levels of crimp and deformation is evident in the migrating yarn paths, along with a reduced through-the-thickness yarn inclination angle approximating 30°.

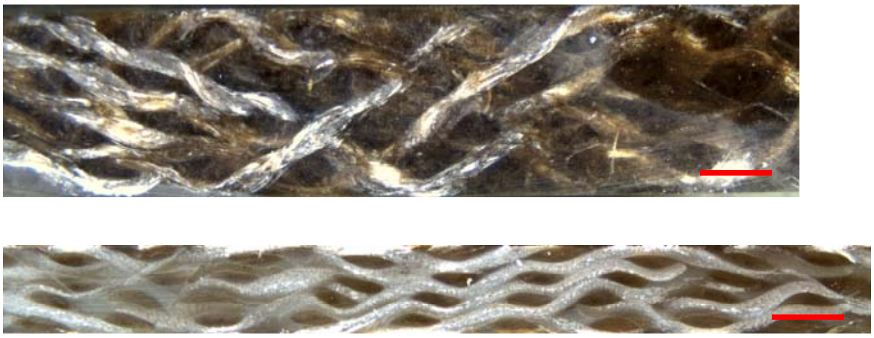

Fig 3: OM micrographs of angle interlock composites: (top) Flax/epoxy composite, (bottom) Viscose rayon/epoxy composite (scale bar = 2 mm)

Evidently, the handle and behaviour of the yarn type chosen and the distortion caused during the weave process are largely responsible for the deformation of yarns within the composite. Although achieving promising performance results, the viscose rayon yarn may be more suited to an orthogonal type architecture where interlacement and migration of yarns is kept to a minimum. If flax is used, a heavier preform that would possess an increased off-the-loom V_f would be required to produce a higher composite V_f with this angle interlock design.

The 3D flax/epoxy composites demonstrated reasonably good flexural performance in comparison to the viscose rayon/epoxy composites relative to their low V_f. These results compare favorably with other work conducted in flax soy resin composites by Huang [25] where unidirectional yarn composites possessing 48% V_f obtained values of 82-117 MPa and 4.7-7.6 GPa in flexural stress and flexural modulus respectively. Huang also reports that flax fabric reinforced composites with 4 layers of laminate ply and possessing a 43% composite V_f obtained flexural stress values of 20.9 – 25.2 MPa in the warp orientation and 0.7– 1.29 GPa in flexural modulus.

Higher V_f of 27-31% were achieved by Liu and Hughes [26] for 2D epoxy/flax laminates. They found that a high V_f was a key driver in delivering improved fracture toughness and also concluded that with superior yarn and textile design a better overall balance of stiffness, strength and fracture toughness is achievable. Another example in this area includes the work done by Williams and Wool [27] who found values of 64MPa and 4.2GPa for 34% V_f non woven flax/soy-oil based composites. Wool [13] states that in a hemp reinforced composite properties possessing a 24% V_f, a tensile modulus of 4 GPa can be achieved. A glass fibre laminated composite using bio-based resin and possessing a 45% V_f was found to achieve flexural strength of 260 MPa and flexural modulus of 11.3GPa. Results from Oksman [14] and Heijenrath and Peijs [17] state flax composite mats with a 47% V_f can obtain similar stiffness to glass fibres using epoxy and polypropylene matrix but with reduced strength.

In this instance, viscose epoxy and viscose furan composites with a much reduced composite V_f, have reached 65% and 50% respectively of the properties of glass fibre composites. This provides significant motivation to generate a full range of tensile, flexural and toughness data from viscose composites to build and improve upon these initial findings.

CONCLUSIONS

Modified Dobby and Jacquard technology can produce orthogonal, angle interlock and near-net shaped woven prototypes. Preforms and composites with volume fractions in the range of 20-40% can generate significant mechanical property data and prototypes using flax and synthetically modified natural fibres. The use of viscose rayon negates some of the disadvantages of flax fibres such as batch variability, need for alkali or enzyme pretreatment and global sourcing in small quantities at more desirable cost. Finding an optimum fabric architecture and fabrication process suited for viscose rayon preforms is crucial to alleviate levels of yarn distortion experienced within the composites. Flax composites demonstrate a robust yarn that resists deformation but requires a higher density preform if performance is to improve.

Epoxy and furan resin systems were utilised successfully in the production of VARTM processed viscose composites. Flexural strength and flexural modulus results are encouraging when compared with leading non-woven research with viscose rayon/furan composites comparable to glass/furan composites. The flax/epoxy results were achieved without any treatment to the yarns to improve interfacial properties between yarn and matrix which remains a key obstacle to further engineering composite usage. Achieving a high V_f is another important issue for flax NFC's.

Work is underway to produce thicker, higher volume fraction composites to compare 3D woven natural fibre with 3D woven glass fibre composites.

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Some of the 3D woven material discussed in this paper has 'patent pending' status.
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