MAGNETISABLE MELT-SPUN FIBRES PRODUCED AS LIQUID-CORE HOLLOW FIBRES: DEVELOPMENT OF FIBRES AND EFFECTS OF MAGNETISABILITY

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ABSTRACT

Magnetisable fibres, among others, can be used for targeted fibre arrangement or fixing by use of a magnetic fields, antistatic properties or shielding against electromagnetic fields. In some cases the application requires to keep the functional, magnetic component separate from the environment. Liquid-filled hollow fibres are a promising candidate for this purpose, as the fibre hull material can be chosen to keep optimal interaction with the surrounding environment, while the liquid-phase allows for introduction of large fractions of fillers. In this study, a novel strategy to achieve magnetisable fibres was investigated. By melt spinning, hollow polyethylene fibres were produced and in situ filled with a liquid containing iron oxide particles. As reference, polypropylene monofilaments were melt spun, where the iron oxide particles were compounded into the polymer prior to melt spinning. Both processes were successfully implemented, however in the first strategy the ferrofluid caused deficient process robustness due to nozzle clogging and thermal instability. For rough access of the achievable magnetisability, hollow fibres were filled manually with a ferrofluid. To evaluate magnetic functionality, a custom-built measurement unit was developed for quantifying the magnetic attraction force of fibre samples. The measured magnetic detachment forces of the ferrofluid-filled samples were 17.0 ±0.8 mN and therefore in the range of the monofilaments, where values from 11.4 ±0.7 mN to 26.6 ±1.8 mN were measured, depending on fibre diameter and iron content.

KEYWORDS

Melt spinning; Hollow fibre; Low-pressure filling; Magnetisable fibres; Magnetisability measurement.

INTRODUCTION

Liquid-filled hollow fibres are a promising candidate to introduce special functionalities into textile products, where the functional component is kept separate from the environment within the fibre core. This way, the fibre hull material can be chosen to keep optimal interaction with the surrounding environment. This opens a wide range of possible future applications. Examples are:

- Magnetisable fibres consisting of a thermoplastic hull and oil in the core, containing iron particles [1]. These can be used for applications like fibre arrangement or fixing by use of a magnetic field, creation of materials with antistatic or magnetic properties, shielding against electromagnetic fields etc [2]. Furthermore, iron containing fluids are known for changing their viscosity in magnetic fields [3]. This opens new options for textile sensors or special protective textiles.
- Materials for drug release in medical applications, where slow diffusion through the fibre wall guarantees a long-term release of the active component [4].
- Fibres with immobilized bacteria in the core for e.g. treatment of sewage water, where the fibre hull allows diffusion of the component to be removed by bacterial metabolism into the fibre core [5].
- Shock-absorbing fibres for protective cloth [6], where the energy absorption is realised by the surface friction between the polymer hull and liquid core.

For all these examples liquid-filled hollow fibres potentially give benefits, with the difference in applications requiring either long fibres (just cut after production) or fibres in lengths comparable to classic staple fibres. In any case it is necessary to close the fibre ends to keep the functional liquid separated from the environment.

In general, liquid-filled hollow fibres can be generated using two different pathways. The first of them consists of two steps: first production of hollow fibres and processing them into a textile or composite and subsequently filling the fibres with liquid [7]. For filling

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there are different options, all of them possible at room temperature: passively using the capillary effect, or by pumping systems either applying pressure [8] or vacuum [7] [9] to soak the liquid into the fibres. Main advantage of these methods is, that there is no demand on temperature stability of the liquid. This enables e.g. filling of glass fibres (produced at high temperatures) but is combined with the disadvantage of limited fillable fibre length (depending on viscosity, internal diameter, surface tension etc.) [10].

The second pathway was developed at EMPA using a special spinneret, where the liquid is injected continuously into the hollow fibre under high pressure. Crucial parameters of this process, influencing the diameters of fibre core and hull, are the ratio between throughput of molten polymer to liquid and their viscosities [11]. Main advantages of this type of process are the unlimited fibre length and high process speeds as base for economical processing [12].

One particular disadvantage of this process is the necessity of applying high pressure to the liquid, preventing e.g. the pumping of liquids containing living bacteria. For this reason, for the works presented in [5], a modified low-pressure pumping system has been successfully introduced into the process, which is the basis for the study at hand.

This article describes the development of the process to generate hollow fibres in-situ filled with an oil containing iron particles ('ferrofluid') by use of a low-pressure system. There is no established method for assessing the magnetisability of individual fibres commercially available. Thus, a custom-built setup for quantifying the magnetic detachment force of the fibres was developed and used in this work.

For comparison and to benchmark the magnetic performance of the liquid-filled fibres, a reference system based on solid composite fibres was required. For this purpose, polypropylene monofilament fibres with iron oxide particles were produced using established polymer fibre manufacturing techniques, which is a proven technology. The typical approach involves integrating magnetic particles – most commonly iron oxides (Fe_3O_4, γ-Fe_2O_3), carbonyl iron, or ferrite powders – into thermoplastic polymers such as polypropylene, polyamide, or polyester prior to extrusion [13-17]. Particle loadings in such fibres typically range from a few percent up to 30 wt%, depending on the desired magnetic response and the constraints imposed by melt viscosity and fibre spinnability [18-20]. These composite fibres have been investigated for various applications including electromagnetic shielding, antistatic textiles, and sensor systems [14-16, 21-29]. Following this established approach, polypropylene monofilament fibres containing iron oxide particles were produced in this study, providing a reference system with well-defined iron content.

MATERIALS AND METHODS

Materials

For the liquid filled hollow fibres, low-density polyethylene (LDPE, LE9168, Borealis, AT with MFR 65 g/10min) was used as sheath material, while refined soybean oil (Chemiekontor, DE, Art. No. 1391-4.5) was used as liquid core. The oil's viscosity is approx. 4.5 mPa·s [30]. Iron oxide particles (Bayoxide® E 8706, Lanxess, DE, average size 30 nm) were mixed in the oil without any additional treatment.

For the monofilaments, polypropylene (PP, Moplen HP561 R, LyondellBasell, NL with MFR 25 g/10 min) was used as fibre and the iron oxide particles were directly compounded in the PP prior to melt spinning.

Methods

Melt-spinning of liquid-filled hollow fibres

Liquid-filled hollow fibres were produced using a modified melt-spinning process with pressureless filling of the fibre core. This enables safely processing of liquids, which are sensitive to pressure changes, i.e. preventing in case of iron particles undesired agglomeration. The key feature of the set-up is a spinneret design in which the polymer melt and the liquid core component remain separated until the spinneret outlet, enabling a merging length of zero. The spinneret (cf. Figure 1) exhibits 18 holes, each with 1.4 mm diameter and internal capillary of 0.8 mm outside diameter and 0.55 mm internal diameter. Subsequently, after leaving the spinneret both components merge, allowing the liquid to be fed without pressure. This approach facilitates the integration of sensitive liquids into the fibre core, which makes it particularly interesting for particle-filled liquids, where the particles could be thermally or mechanically damaged.

A schematic of the process including the applied temperature profile is shown in Figure 2. The polymer (LDPE) was melted in a twin-screw extruder (Leistritz ZSE18) at a screw speed of 100 rpm and conveyed by a gear pump to the spinneret. The extruder zones were operated at temperatures between 110 °C and 130 °C, with a spinneret temperature of 130 °C (see Figure 3). The liquid was transported separately from a reservoir by a peristaltic pump and guided to the centre of each spinneret orifice through coaxially inserted capillaries. At the spinneret outlet, polymer and liquid converged to form liquid-filled fibres, which were subsequently drawn, stretched, and wound. For processing, a spin finish suitable for polyolefins (DURON® OF 4066, Schill+Seilacher, DE) was applied as a 10 wt% solution in distilled water. Multifilament yarns consisting of 18 filaments were obtained at a winding speed of 100 m/min.

Figure 1. Spinneret outlet in detail view during production of liquid filled hollow fibres.

Compounding and melt-spinning of magnetisable polypropylene fibres

Prior to processing, the iron oxide powder was dried for 24 h at 80 °C. Polymer and particles were then pre-mixed before feeding into the extruder. Compounding was carried out on a co-rotating twin-screw extruder (Leistritz, DE) equipped with a 3.5 mm die. The extruder operated at a screw speed of 200 rpm and zone temperatures between 70 °C (feed) and 220 °C (die). The extrusion profile included 13 heating zones, gradually increasing from 70 °C at the feed zone to 220 °C at the die. Compounds with particle loadings of 20 and 30 wt% were produced at a total mass throughput of 2.5 kg/h.

The compounded granules were pelletised and subsequently processed into monocomponent fibres on a melt-spinning line (Fourné Maschinenbau GmbH, Alfter-Impekoven, DE). Fibres were produced at a throughput of 2.1 kg/h and a spin pump speed of 30 rpm.

Optical microscopy

The characterisation of fibre geometry was carried out by light microscopy (Leica MC 120 HD, Leica Microsystems, DE) of fibre cross-sections at randomly selected positions along the multifilaments. For sample preparation, fibres were fixed in a microtome (Figure 4) and cut perpendicular to the fibre axis. The resulting cross-section planes were subsequently examined under bright-field conditions, and outer and inner diameters as well as wall thicknesses were determined from the micrographs.

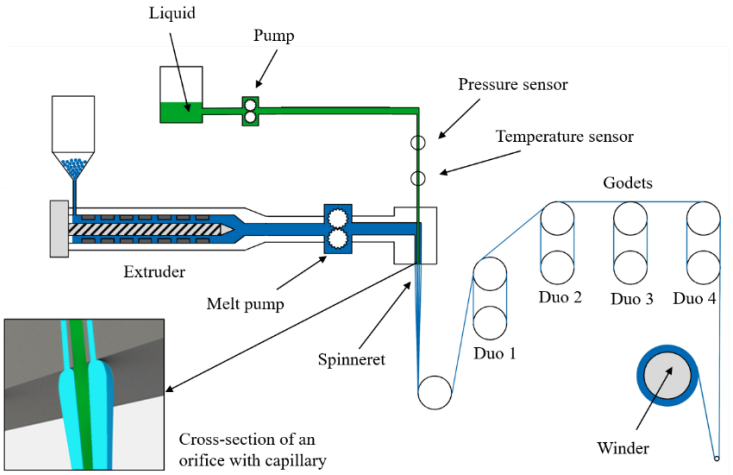

Figure 2. Schematic set-up of the modified melt-spinning process for liquid-filled hollow fibres. The polymer is melted in the extruder and conveyed via a gear pump to the spinneret, while the liquid is transported separately through a hose pump. Polymer melt and liquid core remain separated until the spinneret outlet and only merge after extrusion, enabling pressureless filling of the fibre core.

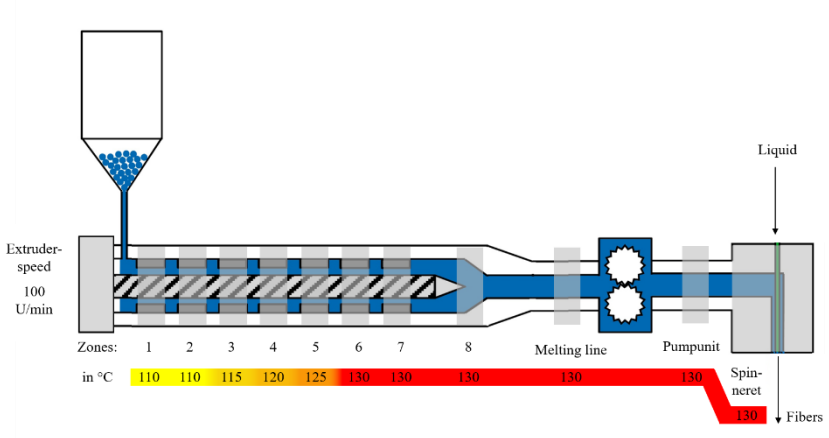

Figure 3. Temperature profile of the extruder used for fibre production. The polymer melt was processed at zone temperatures between 110 °C and 130 °C, with a constant spinneret temperature of 130 °C.

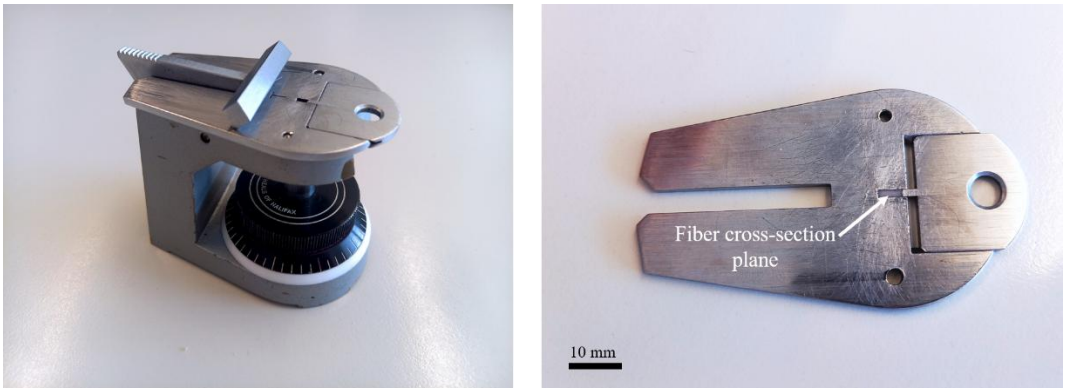

Figure 4. Microtome used for the preparation of fibre cross-sections. Left: microtome device with clamping unit for fibre fixation. Right: positioning of the fibre segment and definition of the cross-section plane obtained for optical microscopy.

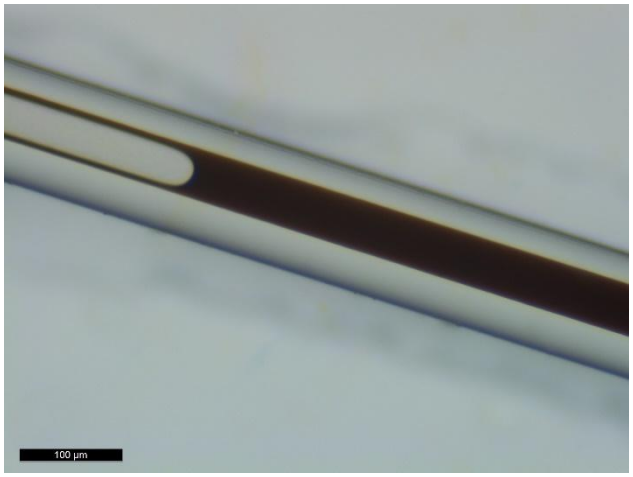

Figure 5. Optical micrograph of the manually liquid-filled hollow fibre.

Magnetisable hollow fibres

LDPE Hollow fibres with diameters of 112 ± 2,3 µm outside and 50 ± 3,1 µm inside were manually filled with a commercial ferrofluid (Laborladen.de, Donaueschingen, DE, Art. No. L10.0071.01000) by capillary forces (cf. Figure 5). This approach was chosen since direct spinning of oil with dispersed iron particles was not stable due to sedimentation and nozzle clogging (see Results section). No quantitative information on the iron content of the ferrofluid was available; the effective iron content of the fibres can be estimated by comparing the results of magnetic detachment to the polypropylene-based compound fibres. This method was suitable to produce enough samples for the magnetic detachment test.

Magnetic characterisation (custom test rig)

In order to quantify the magnetic adhesion force of the fibres, a dedicated test rig was developed (Figure 6). The set-up consists of a glass-fibre reinforced plastic (GFRP) plate on which the fibre bundles are fixed. The plate is equipped with a screw at the bottom side, allowing the specimen to be mounted directly onto a calibrated load cell (KD45 10N, ME-Meßsysteme GmbH, Henningsdorf, DE, Art. No. 14). For all tests, a cable of 36 single filaments single filament was manually wound 40 times around the GFRP plate, ensuring the same number of 1440 filament windings in total for each sample. The tension of manual winding was kept as low as possible to exclude filament elongation. A permanent magnet of 5 mm diameter with an attached string is placed onto the fibre specimen, the sensor is zeroed, and the string is slowly pulled vertically upwards manually by means of a hand crank. The pulling speed was approx. 0.5 mm/s. The force is continuously recorded (sampling rate: 10.64 Hz). The maximum force corresponds to the magnet detachment and is identified by using the @MAX() function in MS Excel. This value is taken as the characteristic magnetic adhesion force. A typical force–time curve is shown in Figure 7. The measurement shows the continuous increase in force as the magnet is lifted, followed by a distinct peak at the point of detachment, which represents the adhesion force. Each measurement was repeated multiple times per fibre type to ensure reproducibility. The load cell is a bending-beam sensor with deformations in the sub-micrometer range, i.e. outside any optically detectable displacement. The measurement therefore directly records the detachment force (peak force), and no displacement information is physically available or relevant for this method. It has to be mentioned, that the measured forces depend directly on magnetic strength and geometry of the permanent magnet used in the set-up. This has to be considered for future developments of this method.

RESULTS & DISCUSSION

Melt-spinning of liquid-filled hollow fibres

Hollow fibres with a liquid core of refined soybean oil were successfully produced using the low-pressure melt-spinning process described above. Figure 8 (left) shows optical micrographs of the resulting multifilaments. The fibres exhibited a homogeneous and continuous liquid core along the filament axis. The outer and inner diameters were uniform over the filament length, and no defects such as ruptures of the fibre wall or leakage of the liquid were observed. The wall thicknesses measured by optical microscopy

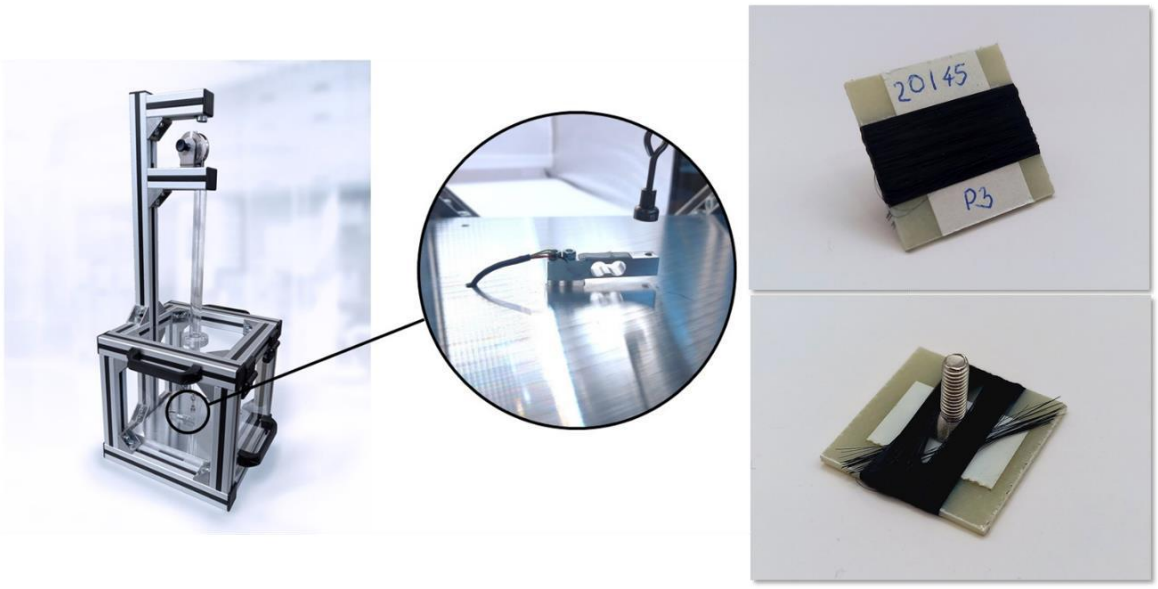

Figure 6. Custom-built test rig for the measurement of magnetic adhesion forces of fibres. Left: overall view of the set-up with vertical frame and integrated load cell. Right: examples of prepared specimens fixed on sample holders for testing (top: fibre bundle, bottom: fibre strands with magnet placed on top, sample size: 15 mm x 15 mm).

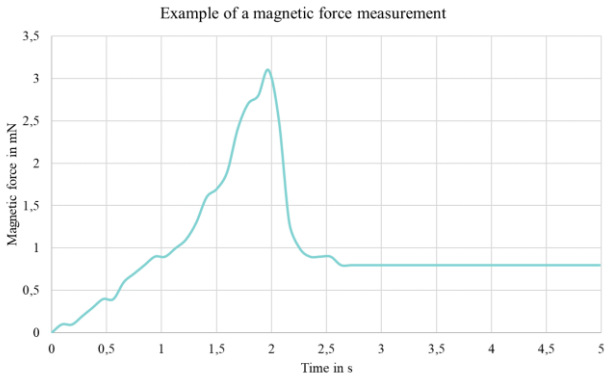

Figure 7. Example of a force–time curve recorded during magnetic adhesion testing. The peak value corresponds to the detachment force of the magnet, which was used as the characteristic adhesion force.

corresponded well to the targeted process parameters, confirming stable processing conditions. The results demonstrate that the low-pressure melt-spinning approach could be transferred in principle, but further development will be necessary for processing magnetisable, particle-containing liquids. Fibres filled with pure soybean oil exhibited continuous, homogeneous cores and stable process conditions, confirming that the liquid dosing system allows reliable core filling without excessive pressure or thermal degradation of the polymer melt.

When the liquid core was replaced by an oil containing dispersed iron oxide particles, the process initially appeared stable, and fibres with a particle-filled liquid core could be produced. However, after short processing time, sedimentation and thermal degradation of the iron-containing liquid occurred, leading to partial clogging of the spinneret and finally to process instability. Moreover, the optical micrographs of these fibres revealed that the particle–oil mixture inside the fibres was not homogeneous. Distinct particle agglomerates (Figure 8, right) can be observed along the fibre axis, indicating that the suspension stability was insufficient under the thermal and shear conditions of the melt-spinning process. The relatively high density and magnetic interaction of the particles likely accelerated sedimentation, while the elevated spinneret temperature promoted changes in viscosity. Similar to observations of Roure & Cunha [31], local concentration gradients occurred, causing clogging of individual capillaries and interruption of the melt flow. Consequently, only short (approximately 10 -15 mm) fibre segments could be obtained.

These findings underline the sensitivity of liquid-core spinning to the rheological and thermal stability of the injected liquid. To achieve stable processing of magnetisable systems, a balance between viscosity, density, and magnetic interaction must be maintained to prevent sedimentation and phase separation. The results further suggest that for applications requiring magnetic functionality, embedding the particles directly in a thermoplastic matrix offers a more robust and scalable approach than dispersing them in a liquid carrier.

Compounding and melt-spinning of magnetisable polypropylene fibres

Magnetisable monofilament fibres with solid particle loading could be produced in a stable and reproducible manner. Both compounds, with 20 wt% and 30 wt% filler loading, showed uniform particle distribution. The magnetisable character of the compounds was immediately apparent, as the pellets were strongly attracted by a permanent magnet (Figure 9, left). Also, both particle loadings could be spun into continuous multifilament yarns without difficulties. No clogging of the spinneret occurred, and

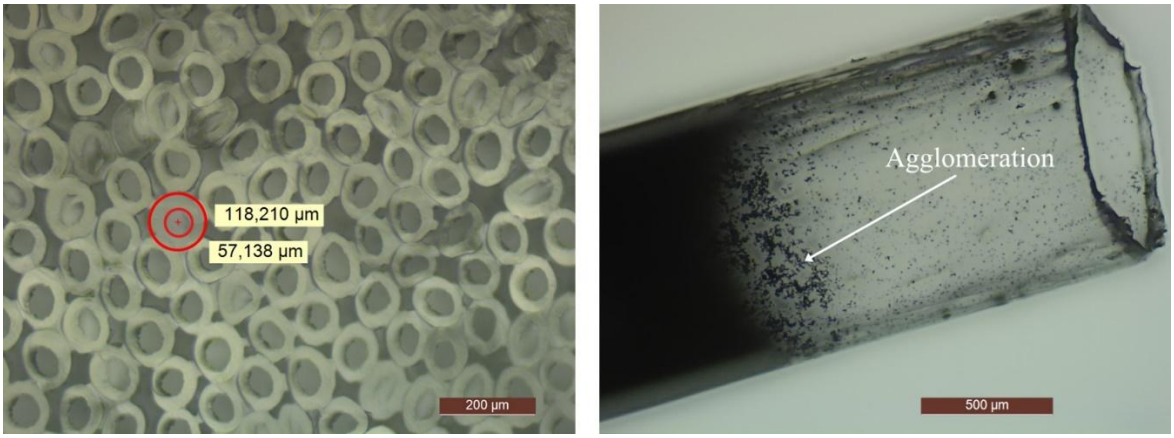

Figure 8. Optical micrographs of liquid-filled hollow fibres. Left: fibres with soybean oil as core material, showing a homogeneous and continuous liquid core. Right: fibre with an iron oxide–oil suspension, revealing inhomogeneous particle distribution and agglomeration within the fibre core.

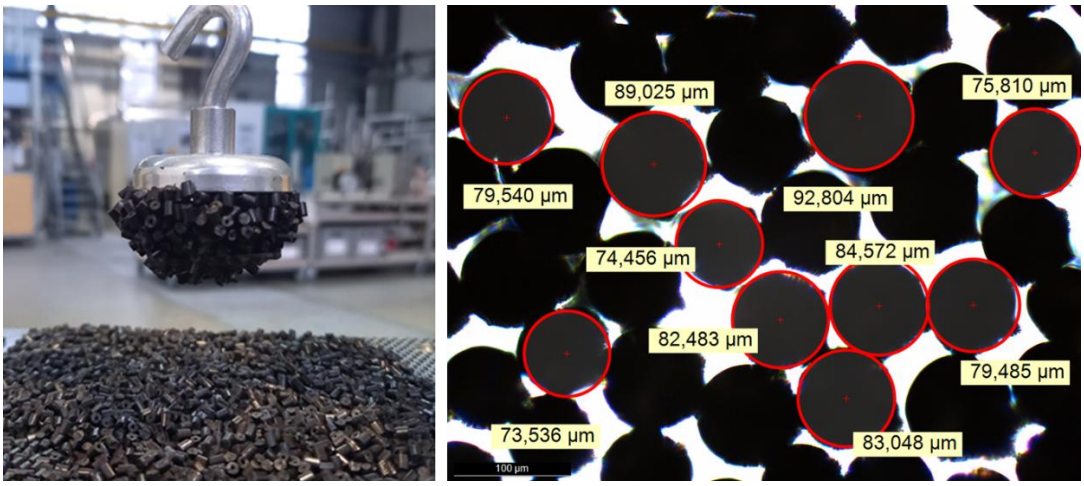

Figure 9. Magnetisable polypropylene compounds and fibres produced from them. Left: polypropylene compound pellets containing Bayoxide E® 8706 nanoparticles (30 wt%) showing strong attraction to a permanent magnet. Right: optical micrograph of cross-sections of fibres spun from the same compound, exhibiting uniform diameters (70–95 µm) and smooth surfaces, indicating homogeneous particle distribution within the polymer matrix.

the filaments exhibited smooth surfaces without visible protruding particles, indicating that the nanoparticles were well embedded in the polymer matrix. In addition, the use of solid-state compounding enabled a higher specific particle loading compared to liquid filling, while maintaining the process stability required for large-scale fibre production.

Optical microscopy of the fibre cross-sections confirmed the uniformity of the produced fibres (Figure 9, right). The fibres exhibited round, homogeneous cross-sections with smooth surfaces and no evidence of particle agglomeration. For fibres with 30 wt% particle content, the measured fibre diameters were in the range of 70–95 µm. These fibres correspond to one of the larger fibre types produced in this study. For subsequent magnetic force measurements, additional fibre types with smaller diameters (30 µm, 45 µm, and 65 µm) were also produced and analysed to assess the effect of fibre thickness on magnetisability.

These results demonstrate that the selected process parameters are suitable for production of magnetisable polypropylene fibres with stable dimensions, well-defined iron content and homogeneous morphology. The resulting fibres were successfully used as reference material in the magnetic detachment force measurements.

Magnetic force measurements

To evaluate the magnetic responsiveness of the fibres, the magnetic attraction force was measured using the custom-built test stand described in the Method section. Six different fibre types were examined, combining three fibre diameters (30 µm, 45 µm, 65 µm) with two particle loadings (20 wt% and 30 wt%). As displayed in Figure 10, the variation of results of repeated measurements on the same test specimen (indicated by the error bars) is in average ±2.89%. This represents the uncertainty of the measurement setup.

The variation among different test specimens is displayed in Figure 11, based on the average of three test per specimen. The box plots represent average value (X), median value (horizontal bar within the box) and first / third quartile (upper and lower box limit).

Again, the variations are small, but as to expect slightly larger than the variations of repeated specimen tests. The number of tests here is too small to allow statistically safe descriptions of the distribution type, but in general it can be stated that the variations are small and in general close to standard distribution. This allows to express the results as average of all nine single values per sample: three specimens, each tested three times.

The measured magnetic forces of all samples are summarised in Figure 12, using the type of evaluation described above. Concerning the mono-component polypropylene fibres, a clear dependence of magnetic force on both particle concentration and fibre diameter is visible. In general, fibres with higher particle loading (30 wt%) generated significantly stronger magnetic forces than those with 20 wt%, confirming that the iron oxide content within the polymer matrix directly affects the magnetic response. The forces measured range from 11.4 ±0.7 mN (30 µm fibre) to 15.6 ±0.7 mN (65 µm fibre) for 20% iron content and from 13.6 ±0.8 mN (30 µm fibre) to 26.6 ±1.8 mN (65 µm fibre) for 30% iron content. For both concentrations, an increase in fibre diameter led to higher measured forces. This effect can be attributed to the higher total magnetic volume per fibre and the resulting increase in magnetic flux density at the fibre surface.

The horizontal green line in Figure 12 represents the magnetic force (17.0 ±0.8 mN) measured for a manually ferrofluid-filled hollow fibre. Compared to the different mono-component fibres serving as reference, this would represent a fibre with 30% iron loading at the diameter of 41 µm. The results demonstrate that magnetisable liquid-filled hollow fibres can achieve equal magnetic forces compared to the solid particle-loaded fibres, while separating the magnetisable particles completely from the environment.

The magnetic force measurements confirmed that the magnetisable polypropylene fibres developed in this study exhibit a clear dependency of magnetic response on both fibre diameter and particle loading. Fibres with higher particle contents (30 wt%) showed distinctly stronger magnetic attraction than those with 20 wt%, which can be attributed to the higher proportion of magnetisable material and the correspondingly stronger interaction with the external magnetic field. In addition, fibres with larger diameters generated higher magnetic forces, indicating that the total magnetic volume per fibre plays a decisive role in the observed behaviour.

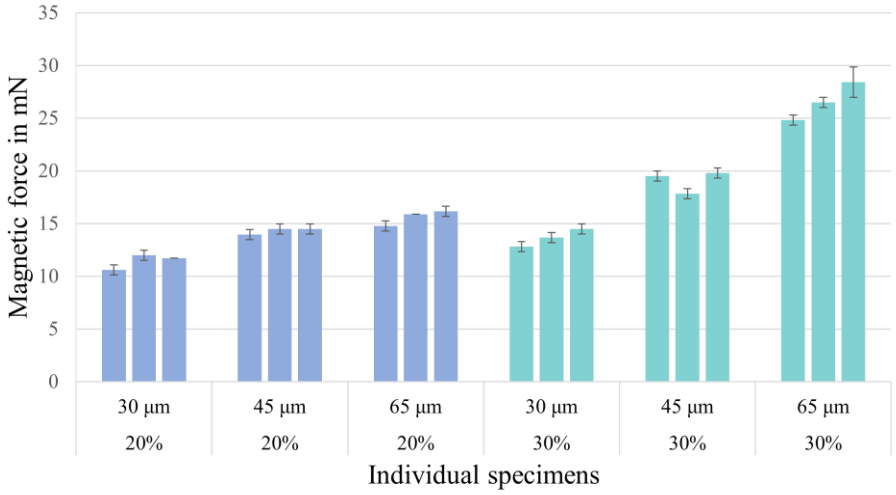

Figure 10. Comparison of the magnetic force of all individual specimens indicating the reproducibility of mesurements.

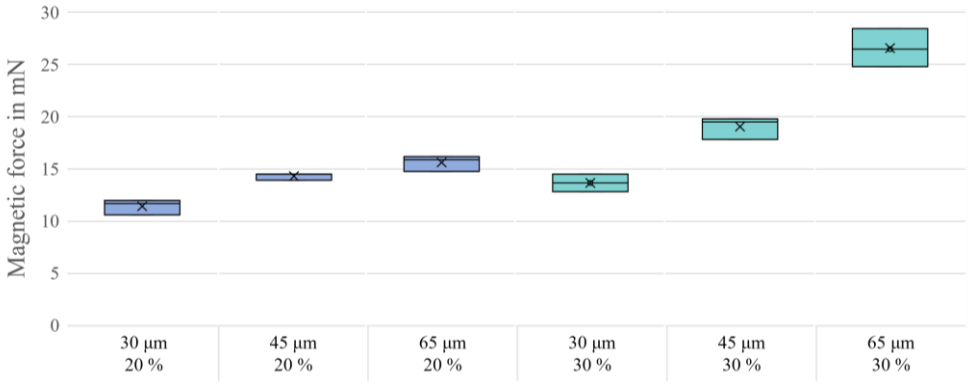

Figure 11. Comparison of the magnetic force of the test specimens per fibre type, indicating the influence of specimen quality.

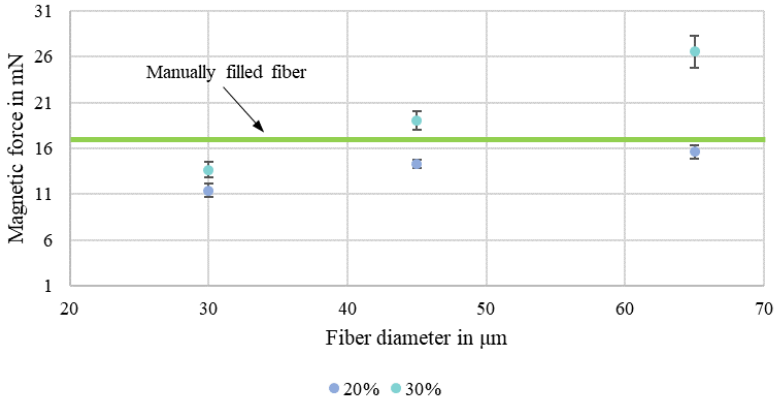

Figure 12. Magnetic force of magnetisable polypropylene fibres as a function of fibre diameter for 20 wt% and 30 wt% particle loading. Error bars represent the standard deviation from three repeated measurements. The green line indicates the magnetic force of a manually ferrofluid-filled hollow fibre used as reference.

A major methodological outcome of this work is the successful development of a dedicated test stand for measuring magnetic attraction forces of individual fibre samples. The system provided reliable, reproducible, and sensitive measurements, enabling a direct comparison of fibres with different compositions and geometries. The obtained data demonstrate that even small differences in fibre structure and particle loading can be quantified with high accuracy.

Overall, the results demonstrate that the magnetisable compound fibres, in combination with the newly developed measurement setup, offer a powerful and reproducible system for studying the magnetic behaviour of polymer-based fibres. Nevertheless, it has to be mentioned, that these results represent a proof-of-concept for the quantification of this type of measurements. For higher statistical safety, influence of the permanent magnet and reproducibility on different set-ups additional research will be necessary.

CONCLUSIONS

In this work, the previously developed melt-spinning process for liquid-filled hollow fibres was adapted and extended to enable the processing of particle-containing core liquids. The process enables the co-extrusion of polymer melts and liquids through a specially adapted spinneret design, allowing the production of fibres with a defined internal liquid core. Using polyethylene and soybean oil as model materials, stable hollow fibres were obtained, proving the general feasibility of the approach.

When magnetisable particles were introduced into the liquid phase, processing challenges such as nozzle clogging and thermal instability of the iron containing fluid occurred. To overcome these limitations, an iron containing fluid with higher thermal stability would be required. Manually filled hollow fibres were instead successfully used as sample material to prove the functionality of magnetisability.

A key outcome of this study is the development of a custom-built measurement unit for quantifying the magnetic attraction force of fibre samples. The test setup proved highly reliable and sensitive, enabling the differentiation of fibre variants based on geometry and magnetic particle content. This novel method allows reproducible magnetic characterisation and establishes a foundation for future systematic studies on polymer-based magnetisable fibres.

Overall, the results demonstrate a new pathway for producing functional hollow and composite fibres with controlled magnetic properties. The combination of innovative fibre manufacturing and the newly developed measurement method represents a step towards the integration of magnetically responsive fibres into technical and smart textile applications.

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