

# EFFECT OF FIBRE LENGTH AND SUSPENSION CONCENTRATION ON ALIGNMENT QUALITY OF DISCONTINUOUS RECYCLED CARBON FIBRE

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## ABSTRACT

Recycling has been widely considered to be one of the crucial requirements for the continuous use and growth in composite material. In this paper, a hydrodynamic alignment process is devised for converting discontinuous random recycled carbon fibre into mats with a highly aligned orientation distribution. The process involves preparation of a fibre suspension stock, fibre alignment via a nozzle, filtration and aligned mat treatment. The aligned mat allows composites with higher volume content to be manufactured at lower moulding pressure thus keeping degradation of fibre length to a minimum. In the past, alignment processes have been focused on fibres shorter than 5mm as they provide good dispersion quality. In the existing work, an attempt was made to use longer fibres in the alignment process as a route to potential enhancement of composite performance. In addition, fibre concentration in the suspension was increased in order to increase the process yield rate. The effect of using longer fibre lengths and more concentrated suspension on alignment quality is reported in this paper. Furthermore, the relationship between nozzle geometry and alignment quality is also investigated. Finally the mechanical properties of a composite laminate made from 3mm recycled fibre is explored.

## 1. INTRODUCTION

The reinforcement potential of carbon fibres is only fully utilised provided the following three main criteria are met; good adhesion with the matrix and sufficient fibre length for efficient stress transfer, a close-packed physical structure for high fibre volume content as well as tailored fibre orientation according to the principal stress directions. These criteria are readily achievable by virgin continuous carbon fibres as their surface has been treated with proprietary sizing, they are available in a variety of tow sizes and can be processed into various form via conventional textile technology. When the composites come to the end of their service life, they are generally shredded for recycling and thus the close-packed tow structure and anisotropic property are removed, producing recycled fibre in a discontinuous form with distributions in length and orientation. Despite insignificant change in surface and mechanical properties, recycled fibre has not been widely accepted by the composite industry due to the difficulty in handling and processing caused by the fluffy discontinuous form. An efficient and cost effective intermediate conversion process is required to improve its physical form to make it more readily adapted to existing composite manufacturing technologies.

Therefore, a range of recycling technologies has been exploited to recover valuable materials from manufacturing scraps and end-of-life items. Generally, these technologies can be divided into four types, mechanical process, chemical process, fluidised-bed process and pyrolysis process [1, 2, 3]. To achieve the recycled carbon fibre with good material properties (high volume fraction), researches have been done many works on developing and improving the alignment processes. Magnetic, electrostatic and hydrodynamic methods have been explored and hydrodynamic process is favoured as it is effective with minimum fibre damage [4]. Explosives Research and Development Establishment (ERDE) developed a breakthrough hydrodynamic process which can be classified as three basic processes: extrusion, filtration and centrifugal process [4]. Relying on its excellent processing properties and high-quality alignment of products, centrifugal process received more attention.

Furthermore, a novel fibre alignment process has been developed at the University of Nottingham which is originated from the ERDE's centrifugal process. In this process, recycled fibre suspension flow through a nozzle and deposit on a nylon mesh which is attached on a rotating drum with vacuum suction plate. And liquid could be removed by the dewatering process combined by the centrifugal force and vacuum suction. In addition, this process has been improved to achieved higher alignment quality for even longer fibres (>5mm).

Usually, fibre suspensions are classified into three concentration regimes according to the fiber volume fraction  $\phi$  and fibre aspect ratio  $r_f$  which is defined as the length  $l$  /diameter  $d$ . If  $\phi < 1/r_f^2$  the suspension is defined in dilute regime and the standard for semi-concentrated regime is  $1/r_f^2 < \phi < 1/r_f$ . For concentrated regime suspension, it is defined as  $1/r_f < \phi$  [5]. In the dilute region, the spacing between the fibres is greater than their length and the fibres are free to move and rotate and hydrodynamic interactions between the fibres are rare. In semi-concentrated suspensions, the spacing between the fibres is less than fibre length but greater than diameter and hydrodynamic interactions are frequent. In the concentrated region, the spacing between fibres is of the order of  $d$  and non-hydrodynamic effects, such as 'physical' collisions between fibres may become important [5]. Thus, even the fibre volume fractions are same, longer fibres could make the suspension more concentrated and harder to disperse.

The aims of this paper are to understand the fundamental science that drives the performance of the Hydrodynamic Fibre Alignment Process; how would the fibre length and fibre concentration of suspension affect fibre alignment quality. Nozzle B, with larger exit open area, was designed to understand the effect from nozzle exit geometry on fibre alignment performance. By comparing the data with existing work the improvements of the process could be proved. In addition, bending tests were performed to show the mechanical property of composite laminate manufactured by 3mm recycled fibre.

## **2. EXPERIMENTAL METHODS**

### **2.1. Materials**

Tenax®-A HT C124 carbon fibre staples coated with a water-soluble sizing, was supplied by Toho Tenax Europe GmbH. The staples length were 3mm, 6mm and 12mm and individual fibre nominal diameter is 7 $\mu$ m. Fibre sizing was removed by placing the staples inside an ashing furnace at 550°C for 15mins. Oleon Glycerine 4810 with glycerol content  $\geq 99.5\%$  was supplied by Univar UK. The glycerine was diluted with tap water until a viscosity of around 300mPas was measured at room temperature (15°C) using a Brookfield LVDVII viscometer. The diluted glycerine was used as a dispersion medium.

### **2.2. Manufacture of aligned mat and compaction test**

#### **2.2.1. Manufacture of aligned mat**

A hydrodynamic alignment process was developed at the University of Nottingham, UK as shown in

Figure 1. During the alignment process, discontinuous fibre dispersion is pumped to a pressure pot and is pressurized to create a consistent flow to a concentric conical nozzle that is located above a rotating drum. A nylon mesh positioned inside the drum is employed as a filter to separate fibre from dispersion medium. Vacuum suction can be applied under the mesh to increase the dewatering rate. The nozzle is fixed to a linear actuator and continuously moves forward and backward to create a certain width fibre mat. The filtrated glycerine is separated from air by a cyclone and is pumped back to a storage tank for reuse. Once the required veil areal density has been met, the slurry deposition process will be halted. The mat is later washed with warm water to remove the remaining dispersion medium and then coated with an epoxy based binder.

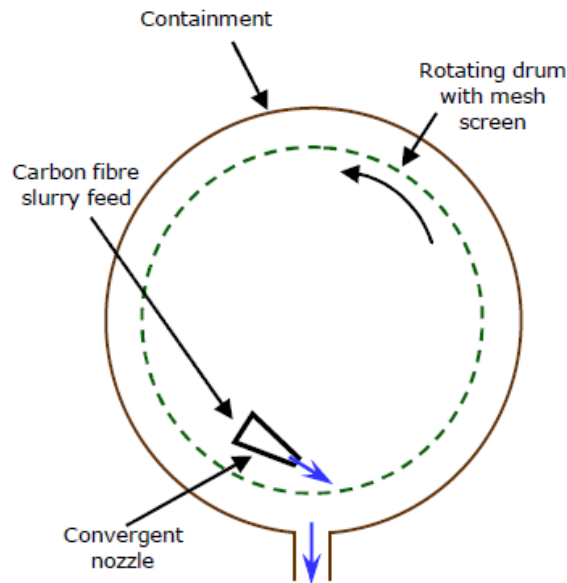


Figure 1: A schematic representation of the fibre alignment rig

In this work, the aligned mats were produced by discontinued virgin fibre with different fibre length and the suspension also contained different fibre concentrations. Details of the factors in experimental design are obtained in Table 1. Furthermore two nozzles were used in order to examine the effect of nozzle exit geometry on fibre aligning quality. Details of the nozzles are given in Table 2. The dimensions of the aligned mats were 115mm in width by 900mm in length.

Fibre Length	Fibre concentration in slurry	Nozzle Exit geometry	Areal density, gsm
3mm	0.1%	Nozzle A	45.58
		Nozzle B	63.18
6mm	0.1%	Nozzle A	62.01
	0.15%		72.22
12mm	0.05%	Nozzle A	51.64
	0.1%		42.7

Table 1: Experimental factors

Nozzle Type	Inlet diameter, mm	Exit area, mm <sup>2</sup>
Nozzle A	25	0.126
Nozzle B	25	3.145

Table 2: Nozzle details

### 2.2.2. Dry mat compaction test

A compaction test rig for measuring carbon fibre mats thickness and voids content (used to determine the average fibre volume fraction) under different compression pressure is shown in Figure 2. The rig essentially contains two flat platens; the bottom platen is fastened to the base of a 5969 universal testing machine from Instron; the upper platen is connected to a 50kN load cell, which is mounted to the machine crosshead. System compliance of the entire rig was first determined by closing the two platens together at a crosshead speed of 1mm/min until a compression pressure of 10bar was registered by the load cell. The crosshead location was automatically logged by the machine's control software during the test. The data was used to measure the distance between the two platens. Two linear variable differential transformers (LVDTs) were mounted on the top platens as

well to provide further readings on the distance between the platens. To measure the thickness and voids content of mats, test specimens of diameter of 50mm were obtained from stamping carbon fibre mat using a cutting template. Each mat was weighed before the test. Then 4 samples of 50mm disc were stacked together and carefully transferred to the bottom platen and were compressed at 1mm/min until 10bar pressure was attained. The thickness and void content of the mats were determined by taking average of the readings from the two LVDT units. The thickness data used in calculation are all measured with dry fibre mats.

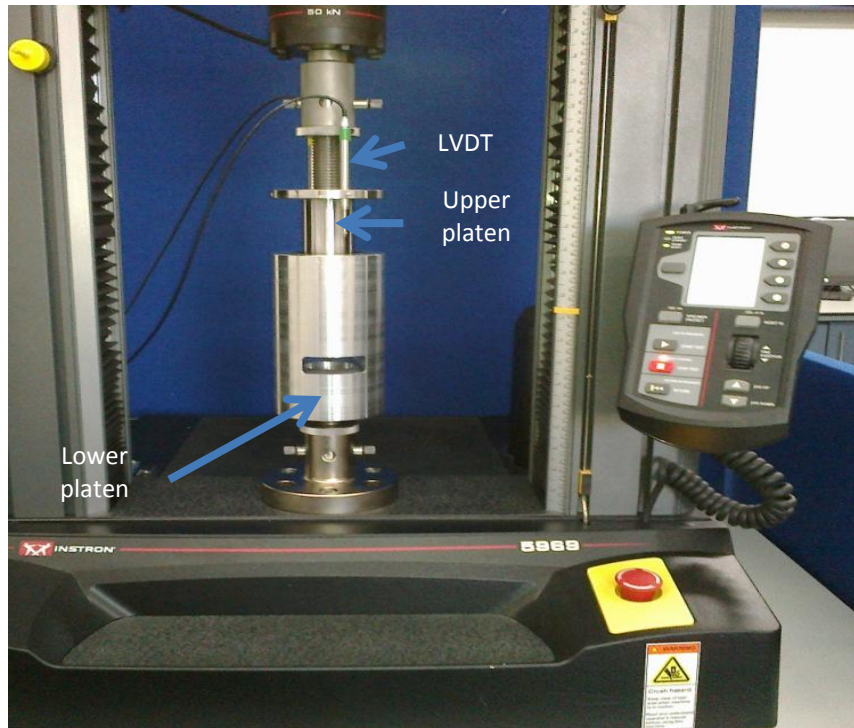


Figure 2: Compaction testing setup for dry mat

## 2.3. Manufacture of composite laminate and Flexural testing

### 2.3.1. Manufacture of a composite laminate

Recycled Toray T800 carbon fibre of average length of 3mm, supplied by ELG Carbon Fibre, UK, was used for making 35gsm aligned mats. There were 61 layers of the aligned mats interlaced with 9 layers of 135gsm MTM57 epoxy resin film, supplied by Cytec Industrial Materials, UK, to produce a laminate with fibre volume of 60%. UK. The materials were vacuum bagged to remove trapped air. Three cycles of 100bars compaction at room temperature were applied. The compacted materials were then moulded at 120°C, 80bar pressure, for 1 hour. Finally, it was cooled down to around 30°C under pressure before demoulding. The moulded laminate's thickness was 2mm.

### 2.3.2. Flexural test on composite laminate

Bending tests were performed on a Hounsfield HTE S-series H25KS/05 universal testing machine according to BS EN ISO 178 (2003). A 1kN load cell was attached to the crosshead, and a test speed of 1mm/min was applied for all specimens, i.e. at least 4 specimens from each laminate. The flexural modulus was defined at strains between 0.05% and 2.5%. Anisotropic properties of the laminate were tested along and perpendicular to the aligned direction.

### 3. RESULTS AND DISCUSSION

#### 3.1. Compaction test

The hydrodynamic alignment process could convert the discontinued recycled fibre into an aligned form in order to achieve a close-packed structure and to maximize the fibre reinforcing potential. And Figure 3 shows the compaction test results from aligned mats made of discontinued virgin carbon fibre using a 1mm diameter circular exit nozzle (Nozzle A). Included also in the Figure are two benchmarking materials i.e. a random recycled carbon fibre mat of 100gsm and a non-crimp virgin unidirectional fabric of 210gsm to demonstrate compaction performance of the aligned mats. The unidirectional fabric was supplied by Sigmatech and was made of T700SC 12K carbon fibre. The random mat contains fibres in a random and filamentised structure, limiting its fibre volume content to around 16% at a pressure of 10bar. On the contrary, the UD structure contains a closed-packed assembly of fibre and thus much higher volume content is readily attainable at the same compaction pressure, i.e. 67.5%. The compaction tests were repeated twice for each aligned mat for consistency demonstration. Of these five mats, the one with 3mm length fibre and 0.1% fibre concentration has the highest alignment quality as it can achieve 44% fibre volume fraction at 10bar pressure. It is 34.8% smaller and 1.75 times higher than the UD fibre mat and random fibre mat, respectively. When the same fibre concentration was used (0.1%) and 6mm fibre was applied instead, the fibre volume fraction would keep as 44% based on the compaction data. However, if 0.15% fibre concentration's 6mm fibre slurry was used the fibre content dropped to 38% at 10 bar pressure. This is 13% less than the data from 6mm\_0.1% fibre suspension. For 12mm fibre mats, the one made by 0.05% concentration's fibre suspension could achieve 40% fibre content at 10bar. On the other hand the fibre content dropped to 32% when the 12mm fibre suspension's concentration was increased to 0.1%. This indicates that the alignment quality of longer fibre is more sensitive to the increasing of fibre concentration. And in the dilute regime, there is a little effect from fibre length on alignment quality.

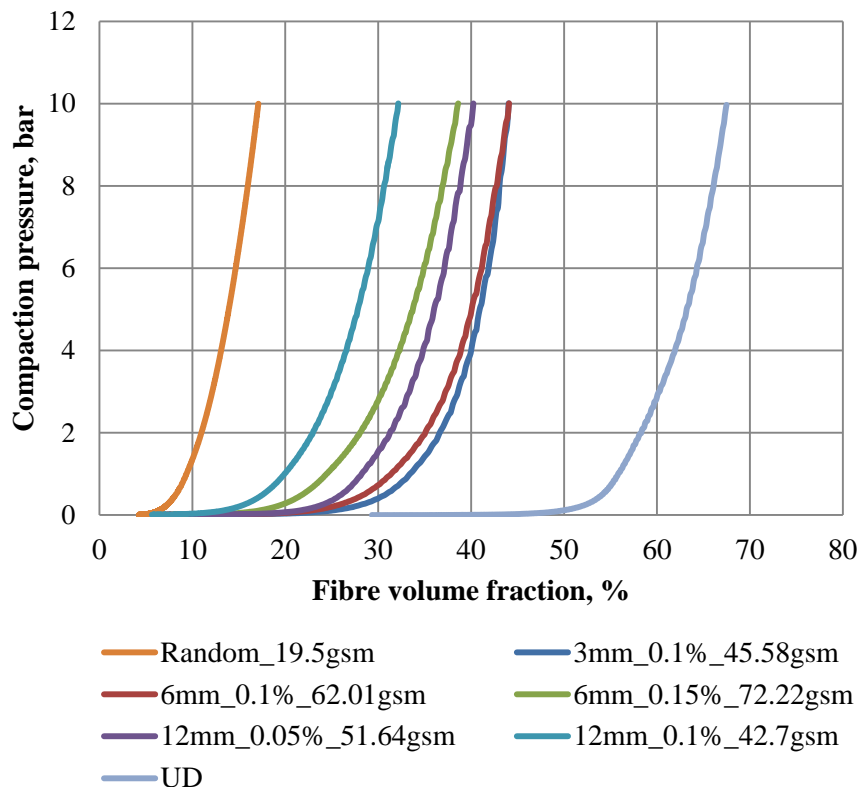


Figure 3: Compaction performance of aligned mats using Nozzle A

In this work, we also looked at the effect of having different geometries at nozzle exist on fibre alignment quality. Normally, Nozzle A was used in hydrodynamic alignment process as it can achieve an isotropic jet. The Nozzle B with larger open area was chosen as it can increase the production rate and reduce the blockage frequency. The larger nozzle exit width could make the suspension jet cover more open area and prevent the air flow get into the mat to damage the aligned fibre orientation during deposition process. Nozzle details are listed in Table 2. Two kinds of mats were produced by different nozzle with 3mm\_0.1% fibre suspension and their compaction performances are compared as shown in Figure 4. The mat made by Nozzle B can only achieve 40% fibre volume fraction at 10bar which is 10% lower than the data show by the mat produced by Nozzle A. Common criterions shared by the two nozzles ware the same pressure setting in the pressure pot and having the same relative speed ratio between drum rotating speed and nozzle transverse speed as 1. This means when the drum rotated one revolution the nozzle moved one nozzle width. The fibre slurry mass flow rate for the Nozzle B was nearly nine times higher, which resulted in a larger amount of liquid medium to be dealt with, compared to the Nozzle A. This created a difficulty in dewatering process, adversely affecting the fibre alignment quality. On the other hand, Nozzle B benefits from an increased production rate and less nozzle blockage.

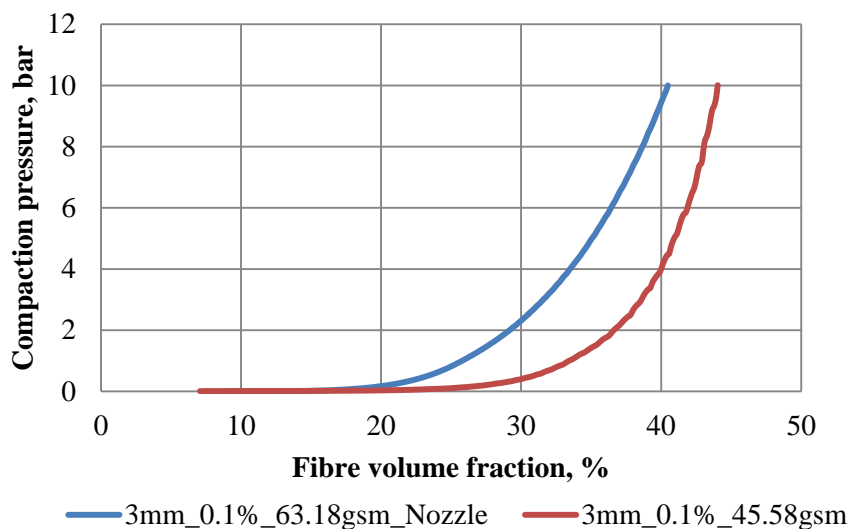


Figure 4: Compaction performance of aligned mats produced using Nozzle B (Blue) and Nozzle A (Red)

### 3.2. Flexural testing on a composite laminate

Results obtained from the flexural test are presented in Figure 5. It can be seen that consistent results were obtained from the test. Table 3 lists the average flexural properties of the laminate. The average breaking stress of those specimens tested along the alignment direction is about a magnitude higher than the cross direction. Similar ratio is observed in flexural stiffness. This clearly indicates good fibre alignment has been achieved using the aforementioned batch process. A micrograph picture of the cross-sectional view of the laminate is shown in Figure 6 and a close-packed fibre structure is evidently shown. Image analysis software was used on Figure 2 and showed that the void content was less than 1% with a fibre volume content of 62%.

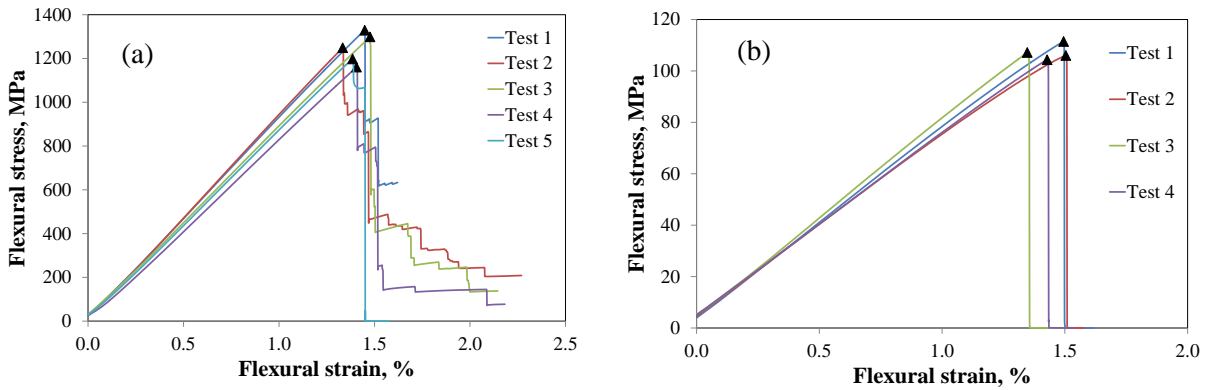


Figure 5: Flexural test (a) along and (b) perpendicular to the fibre alignment

Specimen	Flexural strength, MPa	Flexural stiffness, GPa
Along to the alignment	$1247.92 \pm 68.48$	$81.84 \pm 5.02$
Perpendicular to the alignment	$107.36 \pm 2.07$	$7.23 \pm 0.16$

Table 3: Average flexural properties of the composite laminate

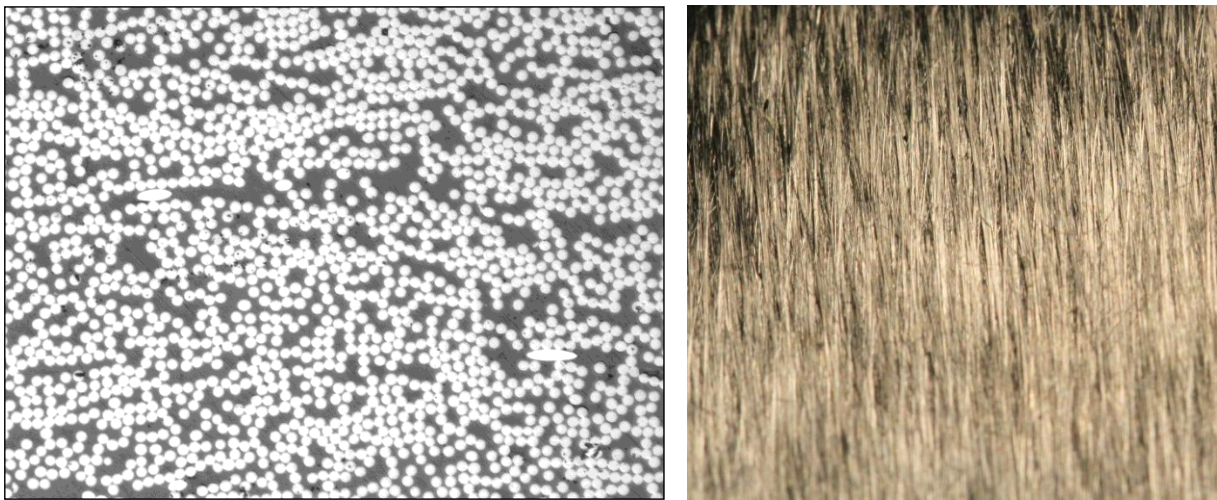


Figure 6: Micrograph of aligned composite laminate (Left), and photo of mat produced (Right)

#### 4. CONCLUSIONS

The developed hydrodynamic alignment has been applied to longer fibre (>5mm) alignment. The effects from fibre length and fibre concentration in suspension were investigated in this paper. The alignment quality of longer fibre is more sensitive to the increasing of fibre concentration. And in the dilute regime, there is little effect from fibre length on alignment quality. The material made by Nozzle B (with larger exit open area) showed 10% less volume fraction than the one made by Nozzle A due to the challenges associated with high rate dewatering, but it also benefits from the higher production rate and less nozzle blockage. Good alignment quality of the composite laminate, made by 3mm recycled fibre, has also been proved by the flexural test and it has a fibre volume fraction as 62% from the analysis of micrograph picture of the cross-sectional view. Future work will also focus on improving the dispersion quality of longer fibre suspension, and increasing the dewatering rate to make better use of Nozzle B.

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