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BALLOT NO. 03-SARG

DRAFT NO. 02

DATE May 16, 2024

WORKING GROUP
CHAIR Martti Tuomisto

SUBJECT CATEGORY
Coating and Graphic Arts (General)

RELATED
METHODS See "Additional Information"

Extensional viscosity of coating color ~~measured~~ evaluated as Euler number using orifice rheometer

(Proposed New Provisional Method)

(Underscores, notes, and strikethroughs show changes from Draft 1)

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1. Introduction

1.1 Coating formulations for paper and paperboard are continuously evolving as new materials, new application processes and new end uses are developed. Even basic formulations (in terms of the simplicity of the formulation or application) can have complex rheological and physical properties that must be understood in order for the coating color to be able to run and perform its intended function.

1.2 A key aspect of the rheological properties is understanding that the shear flow properties (or to use the common term, "shear viscosity") and extensional flow properties (extensional viscosity, also known as elongational viscosity) of the coating color can change as the coating is subjected to different rates of shear and extension as it is mixed, pumped and flowed through pipes, filters and an applicator. Extensional flow occurs when the coating color accelerates or decelerates.

1.3 The extensional viscosity of a fluid can be evaluated using Euler number, which for the flow through an orifice is defined as the extrusion pressure divided by the kinetic energy term. A high Euler number indicates high extensional viscosity. As kinetic energy will always be present, the Euler number will always be 1 or higher.

1.4 The Euler number is calculated by comparing pressure loss to kinetic energy. The entrance pressure loss is also dependent on shear viscosity, which can be problematic when comparing two coating colors with very different shear viscosities.

1.5 Appendix A shows the extensional rate ranges for different coating processes and relevant test equipment.

2. Scope

2.1 This method describes a procedure for measuring the Euler number of coating ~~col~~ colors using a High-Shear Viscometer with an orifice configuration.

2.2 In this method, the extensional viscosity is evaluated by calculating the Euler number when the coating color flows through an orifice at a velocity of 15 m/s.

2.3 The method is applicable for water-based coating colors used in various coating processes such as in blade, rod, and curtain coating. Typically, suitable coating colors have a solids content between 30 % and 80 %, and they contain mineral pigments, synthetic and / or natural polymers and coating additives.

2.4 An Euler number of 1-2 signifies ~~the~~ a lack of extensional viscosity in the sample. Euler numbers of 2-5 signify moderate amounts of extensional viscosity, while numbers over 5 signify high extensional viscosity. Typical extensional viscosity values can be seen in Appendix E.

3. Significance

3.1 Coating color is a non-Newtonian fluid and therefore its rheological properties can be complicated. The key factors influencing a coating color's behavior are the shear and extensional viscosities at strain and extensional rates that the coating is exposed to in the process from make-down to application on a substrate. In coating processes such as blade, rod and curtain coating, ~~deformation~~ extensional rates can reach as high as a ~~million s -1~~ million reciprocal seconds as shown in Appendix A.

3.2 Extensional viscosity is important as it influences the flow and behavior of the coating color in various stages in the coating process. The extensional viscosity refers to the material's ability to resist stretching under stress. During application, the coating needs to form a uniform layer on the substrate. The extensional viscosity can help the coating spread evenly across the substrate surface. It is especially

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important in coating processes where the coating color is stretched or accelerates suddenly, such as in the falling curtain and impingement point in curtain coating. A suitable point to measure the Euler number is around 15 m/s. The substrate in curtain coating can move over 1500 m/min, while the falling curtain typically has a speed of 100 m/min. High extensional viscosity helps the curtain to remain uniform, and it prevents the formation of defects in the coating. Rod coating is another method where extensional viscosity can be of importance. While it is possible to achieve high coat weights with rod metering with a low tube load, this may lead to process instability. On the other hand, when ~~the~~ extensional viscosity is sufficiently high, higher coat weights can be achieved even with increased tube loads, providing a more stable and effective coating process (Kokko, 2001). Extensional viscosity also influences film coating processes, especially at the nip exit, where the coating color experiences stretching perpendicular to the substrate. This stretching can lead to the formation of filaments and result in a misting problem (Yang *et al.*, 2019).

3.3 The orifice viscometer can measure the extensional viscosity as ~~the~~ an Euler number at high flow rates (Appendix B). Extensional viscosity can be attained by using long and coiled-up molecules, capable of extending into the continuous phase over significant distances. The molecules should also form hyperstructures when interacting with pigments or polymeric particles. Finally, the polymer must contain functional groups which interact with the continuous phase, typically water, in which case the polymer backbone ought to contain hydrogen donor and acceptor moieties. The extensional viscosity is also affected by the shear viscosity and solids content (Heinikainen, 2012).

3.4 In filament stretching rheometers a cylindrical liquid bridge is formed between two circular plates. The sample is subjected to strong extensional deformation as the plates are moved apart thereby creating a filament. For sufficiently high viscosity materials, ~~the~~ tensile stress as a function of strain is directly measured, from which the extensional viscosity can then be derived. The CaBER (Capillary Breakup Extensional Rheometer) is a filament stretching rheometer that has been used to measure extensional viscosity for relatively low viscosity materials at low flow rates. During measurement, the formed filament diameter is measured with a laser as a function of time, from which the apparent extensional viscosity can be calculated.

3.5 The MPR (Multi-Pass Rheometer) consists of two pistons and a test section that can accommodate either a capillary or an orifice in between them. By measuring the pressure difference across the test section and the piston movement, it is possible to calculate the extensional viscosity when using the orifice geometry (Mackley and Hassell, 2011).

3.6 A capillary viscometer may also be used to obtain an elongational viscosity-related parameter by analyzing the pressure loss when a fluid enters a capillary (known as the entrance effect). If clearly different viscosities are obtained with capillaries of the same diameter but different lengths, the difference can be attributed to extensional viscosity (Lehtimäki, 2021).

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4. Applicable standards

T582 pm-22 High Shear Capillary Viscosity of Coating Color on Paper and Paperboard

5. Definitions

Euler number is measured as a function of the average velocity of the fluid at the orifice, where the normal range is 0-30 m/s. In this standard, the velocity of 15 m/s is used.

6. Apparatus

6.1 An orifice rheometer is a special case of capillary viscometer designed to measure the extensional viscosity at high flow velocities (Appendix D). Extrusion pressure is measured with a pressure transducer, and the displacement of the sample cylinder is measured with a position sensor. Volumetric flow rate through the orifice is calculated from the displacement of the sample cylinder during the measurement cycle. The flow velocity of the fluid suddenly increases when the sample enters the small orifice. Therefore, a certain part of the extrusion (measured) pressure turns into kinetic energy of the fluid. The Euler number is calculated by dividing the extrusion pressure with the kinetic energy term as shown in Section 11.

6.2 In standard measurements, a metal plate with a 0.5 mm diameter (D) and 0.5 mm long (L) orifice is typically used (Appendix C).However, other diameter orifices can also be used.

6.3 Pressure and position sensors are initially calibrated by the manufacturer. Calibration must be checked once a month as instructed in the user manual.

6.4 The orifices are calibrated with water at 20 bar pressure (the pressure value can be changed). The diameter of the orifice is adjusted until an Euler number close to 1 (±2%) is achieved. It is recommended that after five measurements water is measured through the orifice. If the Euler number of water is out of range of the value 1 (± 2%), the orifice should be recalibrated. In any case, the orifice should be calibrated after fifty measurements. The diameter of the standard orifice must be 0.5 ± 0.01 mm. Otherwise, it needs to be replaced by a new orifice. After each calibration the new diameter is entered in the computer program of the viscometer.

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6.5 A low-speed laboratory mixer, a 300-µm (50-mesh) sieve and a balance with the sensitivity of ±2% are required for the measuring procedure.

7. Reagents and materials

The method can be applied to analyze the extensional viscosity of different paper and paperboard coating

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formulations in blade, rod, or curtain coating processes.

8. Safety precautions

WARNING! – High Shear Orifice Rheometer is meant to measure only non-explosive water-based liquids. It uses high pressure at max. 150 bar.

9. Sampling and test specimens

Maximum volume of a coating color sample is 250 ml.

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10. Procedure

10.1 Cool down / warm up the sample in a water bath to 23 ± 1°C and mix it carefully with a low-speed (< 2000 rpm) laboratory mixer for two minutes.

10.2 Measure the weight of a known volume of coating color with a balance and calculate the density of the sample.
NOTE! Care needs to be taken that during mixing and filtration air is not entrapped as this will impact the density measurements. During the measurement the high pressure will compress the air so that the density used in the calculation of the viscosity could be off.

10.3 Screen 250 ml of coating color through a 300-µm (50-mesh) sieve.

10.4 Make sure the sample cylinder is clean and dry. While washing the sample cylinder, stabilize the cylinder temperature to about 23°C. Pour the sieved coating color into the sample cylinder and place the cylinder into the device.

10.5 Make sure measurement settings are those described in the manual and start the measurement cycle from the control program.

10.6 Read the Euler number from the instrument and save it as raw data.

10.7 After the measurement cycle is complete, take the sample cylinder out and wash it under tap water.

10.8 Allow the sample cylinder to come to ambient temperature before the next test.

11. Calculations

The Euler number can be defined as extrusion pressure / kinetic energy term, and is calculated as:

$$\boldsymbol{Eu} = \frac{\boldsymbol{p}}{\boldsymbol{p_k}} = \frac{\boldsymbol{p}}{\frac{1}{2}\boldsymbol{\rho v^2}} = \frac{\boldsymbol{p}}{\frac{1}{2}\boldsymbol{\rho}(\frac{\boldsymbol{Q}}{\boldsymbol{\pi R^2}})^2}$$

where

Eu = Euler number

p = pressure

v = velocity

ρ = density

Q = flow rate

R = radius

12. Reports

12.1 Report extensional viscosity as Euler number.

12.2 Plot Euler number as a function of velocity.

12.3 Description and density of the sample plus measuring time/date should be printed on the report in addition to shear rate, shear stress and viscosity values.

12.4 Solids content, pH value and Low-Shear viscosity may also be included in the report as they are useful values.

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13. Precision

Round-robin (applicable to T1200) results have shown that the following precision may be expected:

- Repeatability (within a laboratory) = ±5.0%
- Reproducibility (between laboratories) = ±10.0%

The round-robin is based on 3 different laboratories involving 2 coating colors and 5 measurements per sample. The laboratories are equipped with individual devices representing the same model of capillary viscometers supplied by the manufacturer.

14. Keywords

Coating, rheology, viscosity, extensional viscosity, Euler number, orifice, coating color, capillary viscometer, viscometer, rheometer

15. Additional Information

Effective date of issue: To Be Assigned

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16. References

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Kokko, A. (2001). Evaluation of viscosity, elongational viscosity and dewatering on coating colors at high shear rates. Ph.D. thesis, Åbo Akademi University, Turku. Publication Database – http://web.abo.fi/fak/tkf/pap/refbase/ 1 of 1

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Mackley, M.R. and Hassell, D.G. (2011) 'The multipass rheometer a review', Journal of Non-Newtonian Fluid Mechanics, 166(9), pp. 421–456. Available at: https://doi.org/10.1016/j.jnnfm.2011.01.007.

Yang, A. et al. (2019) 'Role of Extensional Viscosity in Paper Coating', Applied Rheology, 21, p. 23607. Available at: https://doi.org/10.3933/ApplRheol-21-23607

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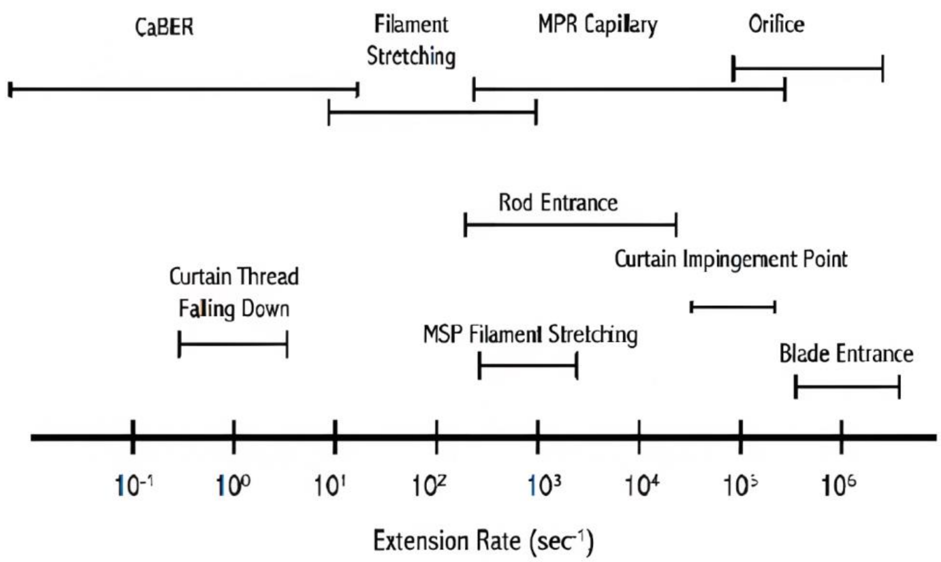
Appendix A: Extension rate ranges for different coating techniques and relevant test equipment.



Source: Yang, A. et al. (2019) 'Role of Extensional Viscosity in Paper Coating', Applied Rheology, 21, p. 23607. Available at: https://doi.org/10.3933/ApplRheol-21-23607

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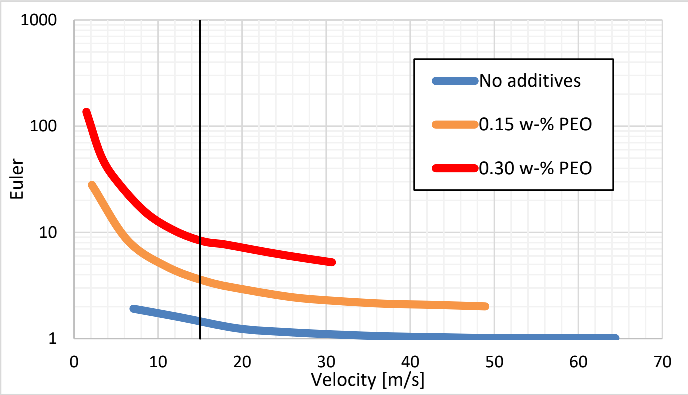
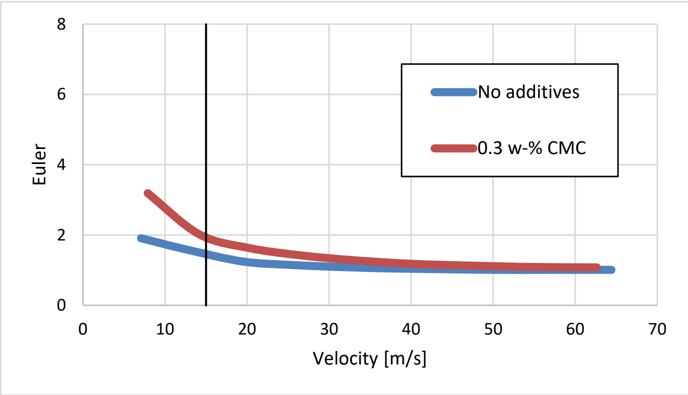
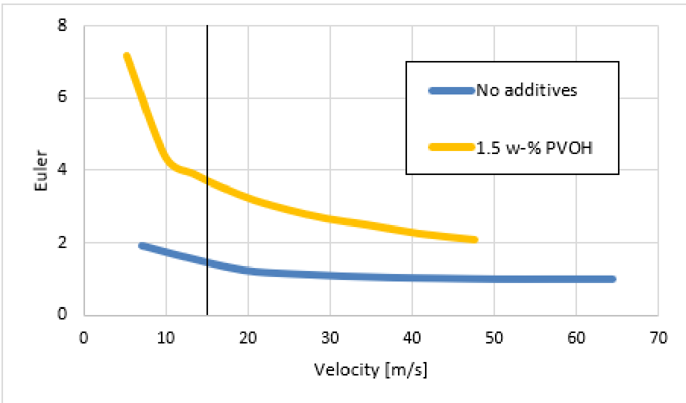
Appendix B: Practical examples of Euler number measurements with orifice geometry. The measured material was a typical water-based barrier dispersion with a 50% solids content.



The addition of high molecular mass Polyethylene Oxide significantly increases the Euler number.
The Euler number rises with higher PEO content.



CMC, a traditional thickener, has no significant effect on the Euler number.



PVOH shows a moderate increase in the Euler number.

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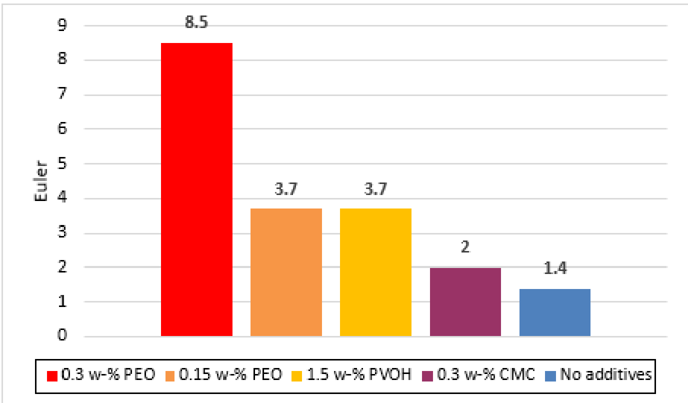

The Euler number of previous graphs at the velocity of 15 m/s.

Appendix C: Photograph of a 0.5 x 0.5 mm orifice.

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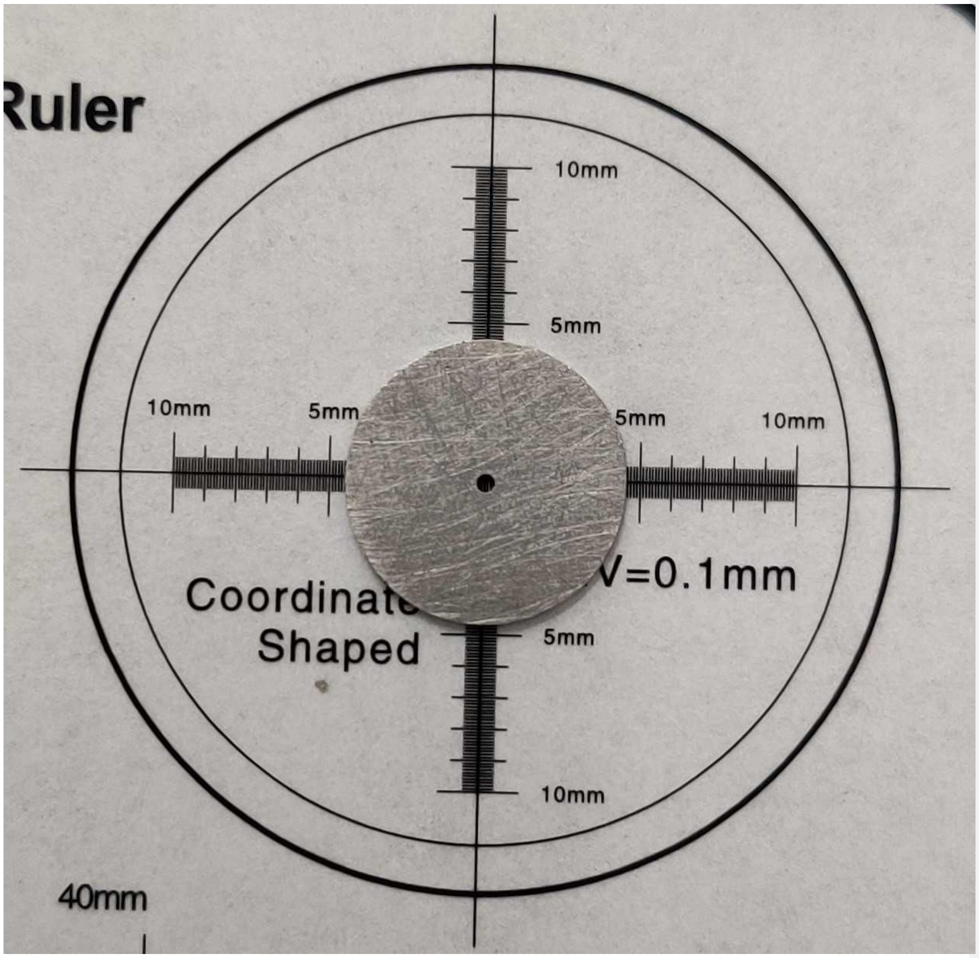

Appendix D: Schematic of High Shear Orifice Rheometer

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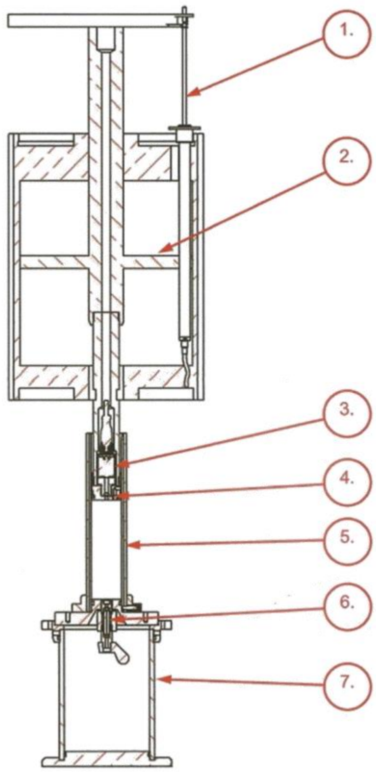

1. **Volume sensor**
2. **Pressure cylinder**
3. **Pressure sensor**
4. **Piston**
5. **Sample cylinder**
6. **Orifice holder**
7. **Removal container**

Appendix E: Typical Euler numbers measured with ACA capillary viscometer using the orifice alternative.

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Application	Solids content	Euler number at orifice flow velocity of 15 m/s
Size / Film press	10…20 %	1-2
Film press	15…40 %	1-3
Film / Blade	40…70 %	1-4
Curtain coating	10…30 %	1-10
Curtain coating	30…60 %	1-20
Special dispersions		1-50

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Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.

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