

## Article Non-Destructive Elemental Analysis of Raster Roller Damage Using X-ray Fluorescence Spectroscopy

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Abstract: Despite numerous methods to optimise their operation and parameters, anilox rolls are subject to rapid wear during use and due to improper cleaning processes. Therefore, regular diagnosis is needed. In this study, X-ray fluorescence (XRF) analysis based on Fe and Cr was used to determine the elemental compositions of raster cylinder coatings. Due to the layered composition of the anilox roll, where  $Cr_2O_3$  coating is applied on the iron core, evaluation of the composition of the roll surface can be used to detection of anilox damage. A portable XRF apparatus was used to identify selected elements even at low concentrations of <1%. In this work, it was proved that XRF can be a preliminary, rapid method for assessing the technical condition of an anilox cylinder. The XRF technique can be safely used in non-destructive chemical analyses of the anilox rollers' condition in flexographic printing technology, and chemical information that aids in their use may be routinely obtained, thus enabling high-quality printing. This is a pioneering study in which the XRF spectroscopy technique was successfully used to anilox roll condition assessment.

Keywords: anilox roll; flexography; ink duct; raster mesh; XRF spectroscopy; elemental analysis



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### 1. Introduction

In flexographic printing, the most important element of the printing machine is the anilox cylinder. Anilox delivers a strictly defined amount of ink from the inkwell to the flexographic printing matrix. This process requires a high level of precision to ensure high and consistent print quality. If the surface of the anilox roll is even slightly damaged, an uneven layer of paint is applied to the photopolymer plate. Therefore, special handling of anilox rolls is extremely important, especially because the purchase cost is very high [1–4]. Anilox rollers can be damaged easily during both the operation process and cleaning before reuse.

In the printing process, anilox gradually wears out because of the surface abrasion of the ceramic or metal layer, which occurs as a result of friction between the raster surface and doctor blade [5–7]. The doctor blade acts as a scraper that removes excess paint above the surface of the inkpot thresholds. Naturally, the scraper constantly rubs against the raster surface, thereby causing wear. The raster grid thresholds widen and lower simultaneously, and a decrease in their depth results in a reduction in the ink capacity of the anilox cells [8–10]. Therefore, the pressure applied by the doctor blade to the anilox roller, referred to as the "kiss take" [11], should be as low as possible. In addition to the pressure and quality of the doctor blade, the wear time of anilox is also influenced by the cleanliness of the ink system and the service culture of the entire system. In parallel, during the printing process, ink dries on the bottom and edges of the anilox cells, which also measurably affects the gradual reduction of the ink capacity of anilox rolls [12–15]. Given that the inkwell capacity defines the volume of ink applied to the product, maintaining a constant ink duct volume is critical for colour reproduction. If the anilox cell capacity

decreases, the amount of ink transferred to the substrate decreases significantly, lowering the optical density of the printout as well as the shades of the printed colours [16–19].

During the use of anilox rollers, in addition to the wear of the ceramic surface, there can be other damage. Damage can occur as a result of impact on the edge of the ceramics, which causes chipping of the ceramics, breaking off of the support sleeve, deformation of the filling layer, and loss of the circular shape of the sleeve, which prevents assembly [20–22]. The formation of peripheral scratches (known as scorings or tiger rings) on raster surfaces is another frequent phenomenon [1,23,24]. Scorings most often occur as a result of the jamming of an element larger than the inkpot thresholds present in the paint between the blade edge of the squeegee and the raster surface [25,26]. This phenomenon causes a scratching effect on the print. If the knife is cleaned immediately, the ceramic coating of the anilox roller is typically not damaged. Otherwise, because of the action of the jammed element on the thresholds of the inkpots, depending on the hardness of the element, a series of thresholds may break, or the thresholds may be lowered to the area where the jammed element rubs against the raster. Breakoffs appear on the surface of the cylinder as thin, sharp scratches. Broken thresholds flow back with the paint again under the doctor's blade, causing the damage to escalate.

This is even more important as the thresholds are often made as thin as possible to increase capacity, which reduces the service life of the raster. Even the printing substrate itself can have an adverse effect on such thin thresholds of the raster mesh. Increased material roughness, such as with recycled paper and cardboard made from recycled pulp containing high amounts of impurities, can also cause accelerated wear on the raster surface. This is worth noting because flexographic printing is often chosen for corrugated cardboard as it ensures very effective printing for uneven substrates [27,28]. As such, there are also no difficulties resulting from transferring paint to substrates with extremely low surface tension, as in the case of plastics (e.g., polyethylene (PE) films and polypropylene (PP) films) [15]. In addition, the increased roughness of paper from recycled pulp means an increase in the porosity of the printing substrate and, consequently, in the ink demand of the substrate, which may also translate into a shorter service life of the anilox roll.

Moreover, an improper cleaning process often damages anilox. The cleaning methods used often do not fully remove paint residues from the inkwells and can also contribute to the delamination of the structure, corrosion, loss of ceramics, and changes in the surface tension of the ceramics [29–33]. To ensure proper paint transfer through the system, it is very important that the ceramic surface provides the lowest possible surface tension and that the surface tension is uniform. Modern anilox rolls are typically made of ceramics with reduced porosity so that the surface tension is relatively low. However, it should be noted that the porosity of the outer ceramic layer may change with each subsequent cleaning, and the surface tension of the raster layer may consequently increase. This results in poorer ink transfer despite the anilox roll regaining its capacity [34–36].

Correct operation and effective cleaning of anilox rolls should, therefore, be priorities for every printing house because they translate into longer raster roller use; that is, better ink transfer increases stability and repeatability during printing and lowers the costs incurred by the printing house. After regular cleaning of anilox rolls, their condition, particularly the condition of the surface responsible for dispensing paint, should, therefore, be evaluated. The wear condition or possible damage to the raster mesh is most often assessed using special microscopes or devices for measuring the capacity of raster grids [37,38]. However, the latter does not directly indicate some forms of damage, such as circumferential scratches. Microscopes are expensive and require appropriate training. Furthermore, microscopy techniques are ideal for laboratory-scale applications, but on an industrial scale, errors in instrument readings may occur owing to vibration, dust, temperature and humidity variations, and operator error. Improper handling can negatively impact the performance of these advanced measuring instruments; therefore, great care is recommended when handling them. Several methods for assessing the condition of anilox that allow quick diagnosis do not require a complicated preparatory procedure or complex service, prompting the authors to research this topic. In the wake of this, in the present work, the authors verified the usefulness of X-ray fluorescence (XRF) spectroscopy for assessing the condition of anilox rolls. However, it must be emphasised that XRF is a complementary method to optical analysis and should be treated as a valuable addition to the microscopic evaluation of anilox roll surfaces.

XRF analysis uses radiation to study the elemental composition of materials [39,40]. The analysis of elements in the examined material is enabled by the behaviour of the atoms when they interact with X-ray radiation. In an XRF spectrometer, a sample is illuminated by an intense X-ray beam, known as the incident beam, and some of the energy is scattered; however, some is absorbed within the sample in a manner that depends on its chemistry. The primary X-ray beam that illuminates the sample is called the excitation beam. The excited sample emits X-rays along a spectrum of wavelengths that are characteristic of the atoms present in the sample. XRF is particularly well suited for investigations involving bulk chemical analyses of major elements (Si, Ti, Al, Fe, Mn, Mg, Ca, Na, K, and P) and trace elements (in abundances > 1 ppm; Ba, Ce, Co, Cr, Cu, Ga, La, Nb, Ni, Rb, Sc, Sr, Rh, U, V, Y, Zr, and Zn). This technique can be applied in many routine, non-destructive chemical analyses. XRF is used, inter alia, in research on igneous, sedimentary, and metamorphic petrology, soil surveys, mining, cement production, ceramic and glass manufacturing, metallurgy, the petroleum industry, studies of various cultural and historical relics [41], objects of art [42,43], and environmental and geological studies (using portable, handheld XRF spectrometers) [44–46].

Due to the layered composition of the anilox roll, where  $Cr_2O_3$  coating is applied on the iron core, evaluation of the composition of the roll surface can be used to determine the condition of the cylinder. For close to 0 values of Fe signals, it shows that the thickness of the coating is above 50 microns (depth of X-Ray penetration into the roll according to XRF device manufacturer). For lower thickness, higher values of Fe signal are observed.

To the authors' best knowledge, this study is the first that applied XRF spectroscopy to anilox roll condition assessment. This analysis made it possible to detect damage by determining the contents of specific elements in the anilox roll coating. The results are discussed in relation to the metal concentrations before and after utilisation of the raster cylinder. Thus, it was proved that XRF can be a preliminary, rapid method for assessing the technical condition of an anilox cylinder.

### 2. Materials and Methods

### 2.1. Material for Research

Several anilox cylinders (in the amount of 6 pieces) delivered by IGT were used for the initial analysis. These cylinders had different rulings ranging from 80 to 180 L/inch. These cylinders were brand-new and coated with chromium oxide (standard  $Cr_2O_3$  coating with a thickness of at least 250  $\mu$ m).

Damaged ceramic cylinders were obtained from a printing house and used in this study. In cylinders with such coatings,  $Cr_2O_3$  forming a ceramic layer was applied to the substrate in a plasma process at a supersonic speed. As part of the analysis, previously used and cleaned cylinders were examined for damage to the ceramic coating.

### 2.2. Research Methods

The analyses were based on the detection of elements, namely Cr and Fe, to determine whether the coating was damaged. As a reference, analyses were performed on previously unused cylinders.

The measurements were performed using a handheld XRF analyser—model X-MET 8000 Expert Geo (Hitachi, Abingdon, UK) with durable IP54-class construction, equipped with a large-area silicon drift detector (SDD) and super-fast BOOSTTM technology electronics, ensuring high sensitivity at lower detection levels. The Mining LE FP testing mode with the full fundamental parameter algorithm was used. The measurement conditions were as follows: circle of 3 mm diameter of the focus point, set using an internal camera

of the device; measuring time of 60 s; distance between the sample and device surface of 100 microns, controlled by the external holder; tube voltage of 50 kV, 4 W. The instrument was a hand-held type, mounted in a specially designed holder so as to keep a constant distance between the sample and the measuring head of the device. Raster cylinders were set facing the X-ray beam and measured at 5–60 s/point to determine the elemental composition. A minimum of three different points for each processed sample were measured, and their averages and standard deviations were calculated and are reported in the figures.

Microscopy images of an anilox roll were obtained through a 3D instrumental optical microscopy technique, using a Keyence VHX 7000 digital microscope (Keyence, Mechelen, Belgium) equipped with VH-Z100UR and OP-72402 lighting ring, with a VH Z500 zoom lens (magnification  $500 \div 5000 \times$ ), and a high-resolution VHX 7020 camera in accordance with the ISO 25178:2016 standard (geometrical product specifications (GPS)). Three parallel microscopy analyses were performed for each of the examined fields of the anilox roll.

#### 3. Results and Discussion

# 3.1. Influence of the Scanning Settings on the Elemental Composition Results for an Anilox Coating

The first stage of the research comprised the verification of the selected spectrophotometer settings. The impact of the scanning time and selected scanning mode on the elemental composition results for the coatings of unused cylinders was verified. Scans were carried out in selective mode, taking into account only Cr and Fe, which allow the assessment of the condition of the coating in terms of damage. It is necessary to clarify that XRF analysis is not adequate for detecting the presence of elements lighter than Na/Mg in the periodic table. Because the ceramic anilox coating is based on  $Cr_2O_3$  and the cylinder spindle is steel, analysis of the Fe and Cr contents is a simple method for detecting damage on the anilox roller surface. In parallel, a full elemental analysis was performed, taking 82 elements into account. The obtained results indicated that the scanning mode used led to no significant differences in the iron and chromium contents in the coating. The Cr content ranged from 61.69% to 63.18%, and with different selected scanning modes, the difference in the content did not exceed 1% (Figure 1). In addition, the selected scanning mode resulted in no significant differences in the iron content (Figure 2). On this basis, elemental analysis of anilox roll coatings using XRF is reliable for calibrating the analyser to selected elements, and full elemental analysis allows the accurate determination of the elemental composition of a raster coating, and thus the detection of damage to its structure.



**Figure 1.** Concentrations of Cr as a function of scanning time determined using portable XRF analyser in selective and full mode.



**Figure 2.** Concentrations of Fe as a function of scanning time determined using portable XRF analyser in selective and full mode.

The scans were carried out at different times to verify the influence of this variable on the results. Figures 1 and 2 clearly show that this variable also had no significant influence on the elemental composition results for a sample. Regardless of whether a sample was scanned for 5 or 60 s, similar Cr and Fe contents were obtained. The negligible Fe content and high Cr content confirmed that the tested anilox had not been used and thus had suffered no mechanical damage to its structure. The chemical analysis data for the major elements of interest (Cr and Fe) are shown in Figures 1 and 2.

### 3.2. Verification of the Suitability of the XRF Technique for Aniloxes with Different Screen Rulings

The raster density, also known as the ruling, is a very important parameter for anilox rollers. It is the number of inkwells per unit length, for example, cm, measured in the direction of the slope of the raster grid. A higher density results in better ink coverage, as it governs the amount of ink transferred to the printing form. As the density increases, the inkwells in the raster structure are packed more closely. Smaller paint drops dry faster, reducing the increase in halftone dots, and thus the reproduced image is more accurate and clearer. The ruling should be based on that of the printing form; the smallest percentage points on the printing form should be greater than those on the inkwells. Otherwise, as a result of the printing form being pressed against the raster surface, the raster points melt in the inkwells. Because of this, their side edges become dirty, and further printing defects, such as splashes, gains, and deformations of the printing points, occur. The screen ruling is also chosen based on the capacity of the inkwells. The only disadvantage of anilox rasters with higher densities is that the size of the inkwells is reduced, which in turn affects their volume; thus, obtaining higher optical colour density becomes more difficult.

Taking into account the significance of the raster density, the usefulness of the XRF method was verified by studying rollers with different screen rulings and inkwell capacities, which are summarised in Table 1.

 Screen Ruling, L/cm
 Inkwell Capacity, mL/m<sup>2</sup>

 80
 24

 90
 20

 90
 16

 120
 12

 140
 8

 180
 4

Table 1. Parameters of raster rollers used for the tests.

Unused rollers were utilised for this purpose to ensure that the inkpot thresholds were not disturbed during the printing or cleaning process; thus, their structures were not damaged, and the capacities did not change. The data obtained during the analyses are summarised in Figure 3. The percentages of both chromium and iron in the ceramic coatings of the aniloxes were almost identical regardless of the inkwell capacity and screen ruling of the cylinder. The Cr content ranged between 62.70% and 63.59%, and the Fe content did not exceed 0.07%, which confirms that the rollers were new and the ceramic coating was undamaged. Based on the obtained results, the XRF technique is suitable for the analysis of anilox with various screen rulings.



**Figure 3.** Concentrations of Cr and Fe as a function of screen ruling determined using a portable XRF analyser.

### 3.3. Analysis of Anilox Damage with Various Scanning Modes (Stationary and Rotating Cylinder)

The next stage of research focused on elemental iron analysis in used aniloxes previously subjected to a cleaning process. The analyses were carried out for rollers in the stationary state when a specimen was rotated, which provided information on the 3D distribution of Fe within the sample. Significantly higher Fe contents were obtained (Table 2) than in the case of unused rollers (Figures 2 and 3), proving that the chromium trioxide layer on the ceramic surface of anilox was disturbed by cylinder utilisation or as a result of improper cleaning.

**Table 2.** Concentrations of Fe determined using a portable XRF analyser in used stationary and rotated aniloxes.

Fe Content, %	
Stationary Cylinder	Rotating Cylinder
8.34	0.88
(9.37) *	(0.97)
6.80	0.82
(7.18)	(0.86)
6.48	0.52
(5.06)	(0.40)
3.50	0.35
(4.63)	(0.46)
9.29	1.45
(3.54)	(0.58)

\* Standard deviations are given in brackets.

The analysis of rotating aniloxes showed that the iron content varied from 0.35% to 1.45%, whereas in the case of new rollers, the content fluctuated around 0% and did not exceed 0.07% (Figure 3). Based on the obtained results, if the iron content in a tested anilox coating exceeds 0.4%, it is highly probable that the cylinder is damaged; if the content is below 0.1%, the ceramic coating of the cylinder most likely has received no damage.

The stationary analysis gave iron contents in the ceramic coatings of used anilox ranging between 3.50% and 9.29%. It should be noted that the iron content is much higher than in the case of a rotating cylinder, and the results have a very large error, as indicated by the very large standard deviations (Table 2). Therefore, evaluation of the states of anilox rollers using the XRF technique is much more reliable and conclusive if performed on a rotating roller, and the analysis covers the surface of the entire roller and not randomly selected areas.

Microscopic analysis of the raster grid surface was also performed to confirm the presence of coating defects detected by the spectrophotometer. The microscopy images show the deterioration of the raster surface in the form of damage to the raster grid thresholds, which have been lowered or even completely worn out, as well as the presence of circumferential scratches visible as wide lines (Figures 4 and 5). The wear of the thresholds, as well as the presence of contaminants at the bottom and around the thresholds, provide extremely important information. Without knowing the capacity of the inkwells, it is impossible to predict how the raster will behave during printing. However, uneven heights and broken thresholds result in inadequate support for the printing point and disqualify such a roller from further use.

At this point, it is worth mentioning that XRF spectroscopy offers many more advantages than disadvantages. The advantages of portable XRF are easy to access and machine manipulation, quick measurements, and quick and automated data evaluation for elemental assays. Thus, XRF spectroscopy can be used even by non-specialist operators in flexographic printing to identify damaged anilox rollers.



**Figure 4.** Images of anilox dot graticule area (**a**) Visible example of (I) damage to inkpot thresholds; (**b**) 3D microscopy image.



**Figure 5.** Various types of permanent damage to the ceramic coating of an anilox roll: (**a**) Example of (II) damage; (**b**) Example of (III) damage; (**c**) Example of (IV) damage; (**d**) Example of (V) damage.

### 4. Conclusions

As previously mentioned, in this study, one of the keys in applying XRF to the assessment of the condition of raster rollers was the analysis of the main elements present in the coatings. Elemental identification with this non-destructive approach provides good information about the condition of the raster surface and thus allows us to distinguish between damaged and undamaged anilox cylinders. The conducted analyses proved that the XRF technique allows flawless initial assessment of the suitability of anilox rollers and the determination and planning of possible regeneration, cleaning, or purchase of new rollers, which in turn translates into significant savings for the printing house. Moreover, the ease and low cost of sample preparation, as well as the stability and ease of use of X-ray spectrometers, give this technique great potential as a major method for determining the condition of raster surfaces based on the elemental analysis of anilox ceramics.

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