Janusz DOMINIK *, *Antoni* SIWIEC **

POLYGRADIENT ELECTROMAGNETIC SEPARATION OF CHLORITES FROM SHALES

UKD 549.623.7:552:[621.928.8:621.318.3

Abstract. The paper presents a method of separation of chlorites from shales by means of polygradient electromagnetic separator. Best results were achieved when grooved plates were placed in the magnetic field and grains above $10\,\mu m$ in size were separated from the suspension. A concentrate with about 80% content of chlorites was obtained.

INTRODUCTION

Chlorites are common components of some sedimentary rocks, but they rarely form pure concentrations. Minerals of the chlorite group show weak paramagnetic properties due to the presence of Fe ions in their crystal lattice. These properties may be utilized for magnetic separation of chlorites from the rocks. The instruments most frequently used for separation of weak-magnetic minerals are Franz isodynamic separator (Flinter 1959; Berry, Jørgensen 1969) or Russian isodynamic separator SIM-1 (Baranow et al. 1966). Separation of fine grains in suspension was carried on earlier by Kac et al. (1962) in the prototype of the SIM-1 separator. These authors were engaged upon separation of artificial mixtures of phlogopite, kaolinite, montmorillonite and nontronite. The SIM-1 separator combined with an ultrasonic device was also used by Babicyn and Ushatinsky (1971) to separate the finest chlorite fractions from kaolinite-illite-chlorite rock.

The separation of chlorites described in this paper was carried out in a polygradient separator designed in the Institute of Mineral Processing of the Academy of Mining and Metallurgy. Control mineralogical tests were performed in the Institute of Mineralogy and Mineral Deposits of the Academy.

* Institute of Geology, Polish Academy of Sciences, Kraków, Senacka 3.
** Institute of Mineral Processing, Academy of Mining and Metallurgy, Kraków, Al. Mickiewicza 30.

DESIGN AND OPERATION
OF THE POLYGRADIENT SEPARATOR

Effective magnetic separation of minerals requires that two basic conditions be satisfied, viz.:

1. Adequate difference in magnetic susceptibility of the minerals being separated,

2. High and heterogeneous magnetic field.

These conditions are rendered by the formula for the magnetic force (F) acting on a grain in the magnetic separator:

$$F = \mu_o \cdot \varkappa (H \cdot V) \cdot H$$

where:

μ_o — magnetic permeability of the vacuum,

$\varkappa$ — magnetic susceptibility of a grain,

H — magnetic field intensity,

V — nabla operator.

When separating weak-magnetic grains, heterogeneity of the magnetic field is of vital importance. Recently, a new type of magnetic separator capable of producing a magnetic field of considerable heterogeneity has been designed. In this apparatus, called polygradient separator (high gradient separator), the spatial differentiation of the magnetic field is attained by placing a set of ferromagnetic balls or grooved plates in the homogeneous magnetic field. The suspension passes through the ducts between the balls or grooves, where the grains are separated into the magnetic and non-magnetic fraction. The former is retained while the latter is removed after passing through the working section of the separator.

Polygradient separators are capable of producing greater magnetic forces that act on mineral grains in the working section than isodynamic separators. A prolongation of the separation time in the latter permits to separate the finest grains; this, however, is attended by a substantial decrease in the capacity. The presented method of separation in polygradient separator is faster but limited to somewhat coarser grain fractions.

SEPARATION PROCESS

Magnetic separation was performed on grey-green and red shales of the Carpathian Flysch, derived from the region of Szczawa. Their main components are minerals of the mica group (muscovite, illite), quartz and chlorite. Feldspars, carbonate minerals and iron oxides are present in subordinate amounts. There are also trace amounts of heavy minerals. Microscopic and X-ray examinations of grain fractions have shown that chlorite forms flakes, 2—120 μm in size. The preliminary preparation of the sample involved ultrasonic disintegration of the rock, removal of free Fe oxides and carbonates, dispersion of clay aggregates with a small amount of NH_4OH, and separation of the size fraction 2—120 μm. The separated fraction was put in the vessel A (Fig. 1) and kept in suspension by constant stirring. The suspension was continuously supplied to the vessel B, placed in the magnetic field. In this plexiglass vessel, there were

balls, 10 and 6 mm in diameter, or metal grooved plates during the successive experiments. Before the balls or plates were placed in the working section of the separator, the induction of the magnetic field was B = 1.15 T. Best results were obtained using the vessel with metal plates. Upon the action of the magnetic field, the minerals with a greater magnetic susceptibility adhered to the plates while the others remained in the suspension

Fig. 1. Polygradient separation unit

1 — electromagnet core, 2 — electromagnetic coil, 3 — control desk, 4 — d.c. generator, 5 — electric wires. A — vessel with suspension, B — vessel with plates or balls in the working section, C — vessel for non-magnetic product
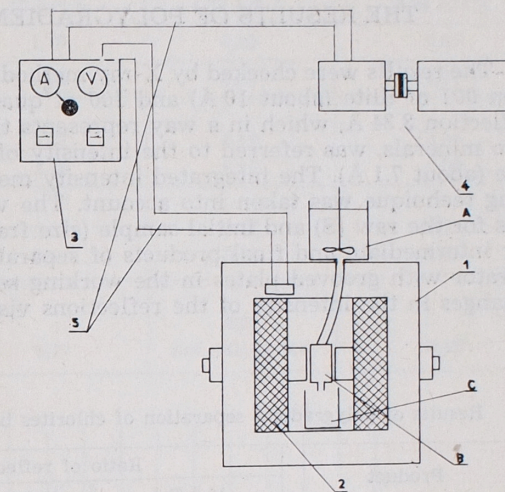
that flowed down to the vessel C placed beyond the range of action of the field. After cutting off the magnetizing current, the magnetic product was washed out of the vessel B by a strong water jet.

Separation was carried out as shown on the diagram in Figure 2. Further separation of the product M_2 did not visibly improve its quality. Since
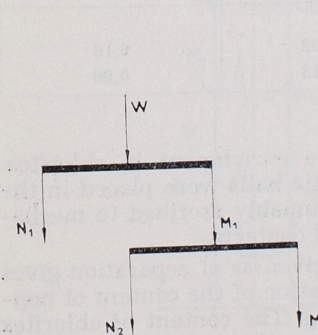

Fig. 2. Diagram of separation with the use of grooved plates

W — initial sample (size fraction 120 — 2 μm); N_1, N_2 — successive non-magnetic products; M_1, M_2 — successive magnetic products
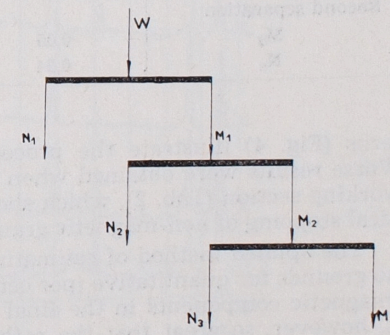

Fig. 3. Diagram of separation with the use of balls

W — initial sample (size fraction 120 — 2 μm); N_1, N_2, N_3 — successive non-magnetic products; M_1, M_2, M_3 — successive magnetic products

the separation was performed with a view to obtaining as pure a chlorite concentrate as possible, the composition of the non-magnetic product was of lesser importance, so this product was not subjected to further separation. Separation with the use of a vessel with metal balls was carried out according to the scheme presented in Figure 3.

THE RESULTS OF POLYGRADIENT SEPARATION

The results were checked by X-ray method. The intensity of the reflection *001* of illite (about 10 Å) and *100* of quartz (4.26 Å) and that of the reflection 3.34 Å, which in a way represents the sum of contents of these two minerals, was referred to the intensity of the reflection *002* of chlorite (about 7.1 Å). The integrated intensity measured by numerical recording technique was taken into account. The values of the mentioned ratios for the raw (S) and initial sample (size fraction 2—120 μm) as well as for intermediate and final products of separation in the polygradient separator with grooved plates in the working section are listed in Table 1. Changes in the intensity of the reflections visible on the diffraction pat-

<div align="right">T a b l e 1</div>

Results of polygradient separation of chlorites by means of grooved plates

Product	Ratio of reflection intensity		
	10 Å/7 Å	4,26 Å/7 Å	3,34 Å/7 Å
S	0,82	0,51	2,96
W	0,57	0,21	1,28
First separation			
M₁	0,20	0,04	0,31
N₁	0,50	0,29	1,72
Second separation			
M₂	0,05	0,02	0,16
N₂	0,24	0,13	0,86

terns (Fig. 4) illustrate the process of sample enrichment in chlorites. Worse results were obtained when ferromagnetic balls were placed in the working section (Tab. 2), which should be presumably ascribed to mechanical stopping of non-magnetic grains at the ball contacts.

The applied method of estimating the effectiveness of separation gives no grounds for quantitative (per cent) determination of the content of non--magnetic components in the final product (M₂). The content of chlorites is, however, so great that the reflections of the other minerals are very weak (Fig. 4) and do not interfere with the interpretation of the chlorite reflections. As appears from chemical analyses, the chlorite concentrate contains about 80% of this mineral; the remaining components (mainly quartz, insignificant amounts of micas, feldspars and heavy minerals) constitute 20% of the sample.

Shale samples subjected to separation may be treated as a mixture of quartz, micas and chlorites. These components differ in magnetic suscepti-

<div align="right">T a b l e 2</div>

Results of polygradient separation of chlorites by means of ferromagnetic balls

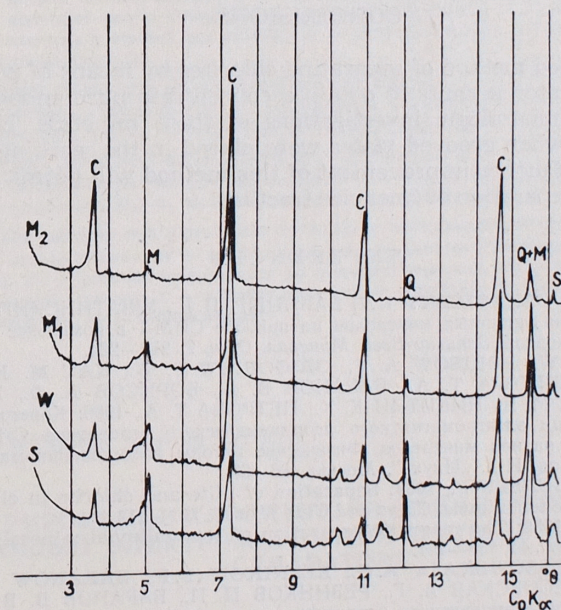| Product	Ratio of reflection intensity		
	10 Å/7 Å	4,26 Å/7 Å	3,34 Å/7 Å
S	1,10	0,45	2,80
W	0,94	0,29	1,77
First separation			
M₁	0,37	0,13	0,86
N₁	1,16	0,48	2,86
Second separation			
M₂	0,22	0,11	0,55
N₂	0,44	0,16	0,84
Third separation			
M₃		0,08	
N₃	0,17	not calculated	0,43

Fig. 4. X-ray diffraction pattern illustrating enrichment in chlorites

S — raw sample, W — initial sample for magnetic separation (fraction 120 — 2 μm), M₁ — magnetic product after the first separation, M₂ — magnetic product after the second separation, C — chlorite reflections, Q — quartz reflections, M — illite and muscovite reflections, Sk — feldspar reflection

bility (e.g., chlorite about $28 \cdot 10^{-6}$ cm³/g, quartz $1 \cdot 10^{-6}$ cm³/g) *. It should be taken into account, however, that the magnetic susceptibility of these components is not constant because the individual minerals of the chlorite group and, to a certain degree, also illite and muscovite may contain variable amounts of Fe ions in the crystal lattice. Vernon (1961) ascertained that the magnetic susceptibility of biotite, connected with the total Fe and Mn content, may vary from 12×10^{-6} cm³/g when the FeO + MnO content is 7.2 w.% to 50.7×10^{-6} cm³/g when the FeO + MnO content amounts to 302 w.%. The magnetic susceptibility of quartz grains may be also modified by the presence of variable amounts of different kinds of inclusions. Thus, even if the optimum experimental conditions are selected, separation of the mentioned minerals in different samples may give somewhat discrepant results, and it is very difficult or even impossible to remove the slight impurities.

The presented method of separation is fast and simple but not effective enough for the size fractions below 10 μm. In that case it is necessary to use method developed by Berry and Jørgensen (1969), which permits to separate chlorite from illite in the size interval 2—4 μm. This method, however, is more time-consuming and less efficient; moreover, the index of contamination with illite, expressed by the intensity ratio of the reflection 10 Å/7 Å is higher (0.12 compared with the value 0.05 obtained when the polygradient method is used).

CONCLUSIONS

The presented method of separating chlorites by means of polygradient magnetic separator permits to obtain a concentrate pure enough to make more precise mineralogic investigations of these minerals. Best results were attained when grooved plates were placed in the working section of the separator. Further improvement of this method will permit, maybe, to use it also for separation of finer size fractions.

REFERENCES

[BABICYN P. K., USHATINSKY I. N] БАБИЦЫН П. К., УШАТИНСКИЙ И. Н., 1971: О сепарации глинистых минералов на приборе СИМ-I в комплексе с ультразвуковой установкой. Записки Всес. Минерал. Общ. 2, 232—234.
[BARANOW W. W., BORISOW A. F., GRIGORIEW P. W., KAC M. Ja., NIKITIN K. K., PIETROWA T. A.] БАРАНОВ В. В., БОРИСОВ А. Ф., ГРИГОРЬЕВ П. В., КАЦ М. Я., НИКИТИН К. К., ПЕТРОВА Т. А., 1966: Конструкция серийного образца электромагнитного изодинамического сепаратора СИМ-I и опыт разделения на нем минералов. Физические методы исследования минералов осадочных пород. Изд. „Наука", Москва. 86—95.
BERRY R., JØRGENSEN P., 1969: Separation of illite and chlorite in clays by electromagnetic techniques. Clays and Clay Min., 8, 201—212.
FLINTER B. H., 1959: The magnetic separation of some alluvial minerals in Malaya. Amer. Miner., 44, 738—751.
[KAC M. Ja., KOSSOWSKAJA A.G., REZNIKOW P.P., BARANOW W.W.] КАЦ М. Я., КОССОВСКАЯ А. Г., РЕЗНИКОВ П. П., БАРАНОВ В. В., 1962: Конструкция электромагнитного изодинамического сепаратора и опыт разделения на нем минералов. Физические методы исследования осадочных пород и минералов. Изд. Акад. Наук СССР, Москва, 118—136.
VERNON R. H., 1961: Magnetic susceptibility as a measure of total Fe plus Mn in ferromagnesian silicates. Amer. Miner., 46.

---

* The data cited after Kac et al. (1962).

Janusz DOMINIK, Antoni SIWIEC

WYDZIELANIE CHLORYTÓW Z ŁUPKÓW ILASTYCH PRZY POMOCY ELEKTROMAGNETYCZNEGO SEPARATORA POLIGRADIENTOWEGO

Streszczenie

Przedstawiono metodę wydzielania chlorytów ze skał typu łupków mułowcowo-ilastych stosując elektromagnetyczny separator poligradientowy. Metoda pozwala na otrzymanie koncentratu o wystarczającej czystości dla wykonania bliższych badań minerałów grupy chlorytów. Najlepsze wyniki osiągnięto umieszczając w polu magnetycznym płyty rowkowane, oraz wydzielając z zawiesiny wodnej ziarna o wielkości powyżej 10 μm. Dalsze doskonalenie omówionej metody pozwoli być może stosować ją także do rozdzielania drobniejszych frakcji ziarnowych.

OBJAŚNIENIE FIGUR

Fig. 1. Zestaw aparatury do separacji poligradientowej
1 — korpus elektromagnesu, 2 — uzwojenie elektromagnesu, 3 — pulpit sterowniczy, 4 — generator prądu stałego, 5 — przewody elektryczne. A — naczynie z zawiesiną, B — naczynie z płytami lub kulkami w przestrzeni roboczej, C — naczynie dla produktu niemagnetycznego

Fig. 2. Schemat stosowany do separacji przy użyciu płyt
W — próbka wyjściowa (frakcja ziarnowa 120 — 2 μm), N_1, N_2 — kolejne produkty niemagnetyczne, M_1, M_2 — kolejne produkty magnetyczne

Fig. 3. Schemat stosowany do separacji przy użyciu kulek
W — próbka wyjściowa (frakcja ziarnowa 120 — 2 μm), N_1, N_2, N_3 — kolejne produkty niemagnetyczne, M_1, M_2, M_3 — kolejne produkty magnetyczne

Fig. 4. Dyfraktogramy rentgenowskie obrazujące wzbogacanie w chloryty
S — próbka surowa, W — próbka wyjściowa do separacji magnetycznej (frakcja 120 — 2 μm), M_1 — produkt magnetyczny po pierwszej separacji, M_2 — produkt magnetyczny po drugiej separacji, C — refleksy chlorytu, Q — refleksy kwarcu, M — refleksy illitu i muskowitu, Sk — refleks skaleni

Януш ДОМИНИК, Антони СИВЕЦ

СЕПАРИРОВАНИЕ ХЛОРИТОВ ИЗ ГЛИНИСТЫХ СЛАНЦЕВ С ПОМОЩЬЮ ЭЛЕКТРОМАГНИТНОГО ПОЛИГРАДИЕНТНОГО СЕПАРАТОРА

Резюме

Описан метод сепарации хлоритов из пород типа алеврито-глинистых сланцев с помощью электромагнитного полиградиентного сепаратора. Этот метод позволяет получать довольно чистые концентраты хлорита,

пригодные для проведения более детальных анализов минералов этой группы. Самые лучшие результаты были получены путем помещения в магнитном поле бороздчатых пластин и при выделении из водной суспензии частиц величиной более 10 мкм. При дальнейшем усовершенствовании описанного метода очевидно его можно будет применять для сепарации более мелких фракций.

ОБЪЯСНЕНИЯ К ФИГУРАМ

Фиг. 1. Комплекс аппаратуры для полиградиентной сепарации
1 — корпус электромагнита, *2* — обмотка электромагнита, *3* — панель управления, *4* — генератор постоянного тока, *5* — электропровода. А — сосуд с суспензией, В — сосуд с пластинками или шариками в рабочем пространстве, С — сосуд для немагнитного продукта

Фиг. 2. Схема сепарации с помощью бороздчатых пластин
W — исходная проба (гранулометрическая фракция 120 — 2 мкм), N_1, N_2 — последовательные немагнитные продукты, M_1, M_2 — последовательные магнитные продукты

Фиг. 3. Схема сепарации с помощью шариков
W — исходная проба (гранулометрическая фракция 120 — 2 мкм), N_1, N_2, N_3 — последовательные немагнитные продукты, M_1, M_2, M_3 — последовательные магнитные продукты

Фиг. 4. Рентгеновские дифрактограммы, показывающие обогащение хлоритом
S — сырой образец, W — исходная проба для магнитной сепарации (фракция 120 — 2 мкм), M_1 — магнитный продукт для первой сепарации, M_2 — магнитный продукт для второй сепарации, C — рефлексы хлорита, Q — рефлексы кварца, M — рефлексы иллита и мусковита, Sk — рефлекс полевых шпатов